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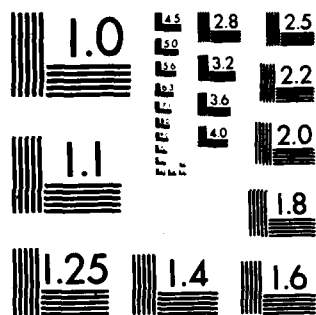
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TECHNICAL REPORT 8215

TERTIARY TREATMENT OF EFFLUENT FROM HOLSTON AAP
INDUSTRIAL LIQUID WASTE TREATMENT FACILITY

II. CORONA OXIDATION STUDIES:
TNT, RDX, HMX, TAX, AND SEX

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AUGUST 1983

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| 1. REPORT NUMBER TECHNICAL REPORT 8215 | 2. GOVT ACCESSION NO. AD-A136877 | 3. RECIPIENT'S CATALOG NUMBER |
| 4. TITLE (and Subtitle) TERTIARY TREATMENT OF EFFLUENT FROM HOLSTON AAP INDUSTRIAL LIQUID WASTE TREATMENT FACILITY II. CORONA OXIDATION STUDIES: TNT, RDX, HMX, TAX, AND SEX | | 5. TYPE OF REPORT & PERIOD COVERED Technical Report September 1981 - March 1982 |
| 7. AUTHOR(s) EDMUND A. KOBYLINSKI, CPT, MSC W. DICKINSON BURROWS, Ph.D., PE | | 6. PERFORMING ORG. REPORT NUMBER |
| 9. PERFORMING ORGANIZATION NAME AND ADDRESS US Army Medical Bioengineering Research and Development Laboratory, ATTN: SGRD-UBG Fort Detrick, Frederick, MD 21701 | | 8. CONTRACT OR GRANT NUMBER(s) |
| 11. CONTROLLING OFFICE NAME AND ADDRESS US Army Medical Research and Development Command ATTN: SGRD-RMS Fort Detrick, Frederick, MD 21701 | | 10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS 62720A 1L162720D048 |
| 14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office) | | 12. REPORT DATE August 1983 |
| | | 13. NUMBER OF PAGES 49 |
| | | 15. SECURITY CLASS. (of this report) UNCLASSIFIED |
| | | 15a. DECLASSIFICATION/DOWNGRADING SCHEDULE |
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| 17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report) | | |
| 18. SUPPLEMENTARY NOTES | | |
| 19. KEY WORDS (Continue on reverse side if necessary and identify by block number) TNT Wastewater RDX Oxidation HMX TAX SEX | | |
| 20. ABSTRACT (Continue on reverse side if necessary and identify by block number) ✓ Destruction of TNT, RDX, HMX, TAX, and SEX in aqueous solution by corona oxidation has been studied in the batch mode. The munition compounds are individually degraded by the Innova process within a 2-hour time period and can be effectively degraded in a multicomponent system. Degradation products were not identified, but evidence is presented for both oxidation and reduction processes. ↑ | | |

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PREFACE

The research reported herein was performed at the request of Large Calibre Weapons Systems Laboratory, US Army Armament Research and Development Command (ARRADCOM), Dover, NJ, under R&D Project No. 1L162720D048, "Tertiary Treatment of Holston AAP Industrial Waste Treatment (ILWT) Effluent," Mr. Bossie Jackson, Jr., project officer, Energetic Systems Process Division. This study is part of the DARCOM Pollution Abatement and Environmental Control Technology Program conducted by the US Army Toxic and Hazardous Materials Agency, Aberdeen Proving Ground, MD. High performance liquid chromatography (HPLC) analyses were performed at US Army Medical Bioengineering Research and Development Laboratory (USAMBRDL) by Mr. Ernst E. Brueggemann, and statistical analysis of data (analysis of covariance) was carried out by Mr. Paul H. Gibbs. Dr. Elizabeth P. Burrows performed and interpreted GC-MS analyses.



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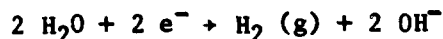
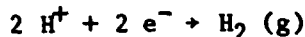
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INTRODUCTION

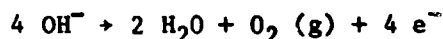
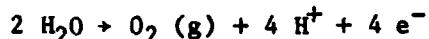
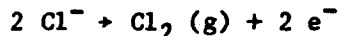
Holston Army Ammunition Plant (HSAAP) will produce 125 million gallons of wastewater per day at full mobilization. The current design of an MCA (Military Construction Army) project for the Industrial Liquid Waste Treatment Facility (ILWTF) at HSAAP does not provide a tertiary mode of treatment for the removal of pollutant chemicals which survive primary and secondary treatment. There is evidence that RDX, HMX, and TNT, as well as biproduct nitramines such as hexahydro-1(N)-acetyl-3,5-dinitro-1,3,5-triazine (TAX) and octahydro-1(N)-acetyl-3,5,7-trinitro-1,3,5,7-tetrazocine (SEX), will survive secondary biological treatment, at least in part, and may adversely affect aquatic organisms in receiving waters. A problem is foreseen in complying with proposed drinking water criteria of 0.049 mg/L for TNT and 0.035 mg/L for RDX/HMX and ambient criteria of 0.06 mg/L for TNT and 0.3 mg/L for RDX/HMX for protection of aquatic life, based on studies by USAMBRDL.

BACKGROUND INFORMATION

This, the second study in tertiary treatment technologies, uses electrolysis to alter the five HSAAP munitions wastewater contaminants. Under the influence of an applied electrical potential, oxidation and reduction reactions occur at the electrodes. At the negative electrode (cathode) reduction reactions occur, such as:



Oxidation reactions occur at the positive electrode (anode) such as:



The reactions are limited only by the concentrations of available reactive species and the ability to accept or release available electrons.¹

Electrolysis is used commercially to produce chlorine, refine metals, electroplate, and treat cyanide wastes. The traditional electrolysis apparatus consists of a narrow cell with the electrodes forming the walls. Various mechanical techniques are used on dilute solutions to overcome slow diffusion rates and improve system performance.¹ Since all reactions occur at the electrodes, the process is limited by the available electrode surface area.

The Innova process uses graphite fiber particles continuously mixed in solution to enhance the removal or destruction of the munition compounds. As the fiber particles move through the electric field, they pick up a charge. Whenever two particles collide, electrons are transported from one particle to the other because of the voltage difference between them. This also occurs at the anode and cathode. As the electrons are transported from one particle to another, the electrochemical reactions occur. In essence, the large number of

colliding particles greatly increases the electrode surface area, resulting in increased reaction rates.² The inventor has presented evidence that the rapid movement and large number of graphite particles within the unidirectional field produce the conditions of high frequency and voltage resulting in corona discharge.

MATERIALS AND METHODS

INNOVA APPARATUS

The Innova apparatus has four major components (Fig 1). The first component is a STACO model EJ401V AC/DC power supply. The second unit is an EMICO^R model RF2 1/4C-2310A DC ammeter. The third unit is an EMICO^R DC model RF2 1/4C-2342 DC voltmeter. The Plexiglas^R tubular reactor, the final component, is 4 1/2 in in diameter and 15 in high. Three platinum anodes are suspended in the center of the column, and a stainless steel mesh cathode cylinder is flush against the outer wall (Fig 2). Air holes are cut into the bottom of the vessel. Air can be provided from any source; a Whisper^R 700A diaphragm pump was used to supply air for agitation. The standard zeta particle (graphite fiber ball) furnished by Innova was used in all experiments.

MATERIALS

Five munitions compounds were used for this study. 1,2,3,4,5,6-Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX) and 1,2,3,4,5,6,7,8-octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX) were provided by HSAAP and were of washed, crude quality (Table 1). Both were air-dried to constant weight before use. 1-Acetyl-1,2,3,4,5,6-hexahydro-3,5-dinitro-1,3,5-triazine (TAX) and 1-acetyl-1,2,3,4,5,6,7,8-octahydro-3,5,7-trinitro-1,3,5,7-tetrazocine (SEX) were prepared by SRI International, by methods described previously,^{3,4} and were used as received (Table 1). 2,4,6-Trinitrotoluene (TNT) was synthesized at USAMBRDL and recrystallized from ethanol. The structures for TNT, RDX, HMX, TAX, and SEX are presented in Figure 3.

TABLE 1. CONSTITUENTS OF TEST SUBSTANCES

| Substance | Purity, % (dry basis) | Other Constituents, % |
|-----------|--------------------------|--------------------------------|
| TNT | est. >99 | |
| RDX | 88.61 | HMX, 11.39 |
| HMX | 98.76 | RDX, 1.24 |
| TAX | 99 | |
| SEX | 96.7 | HMX 2.4, DADN ^a 0.9 |

a. 1,5-Diacetyloctahydro-3,7-dinitro-1,3,5,7-tetrazocine.

* Use of trademarked name does not imply endorsement by the US Army, but is used only to assist in identification of a specific product.

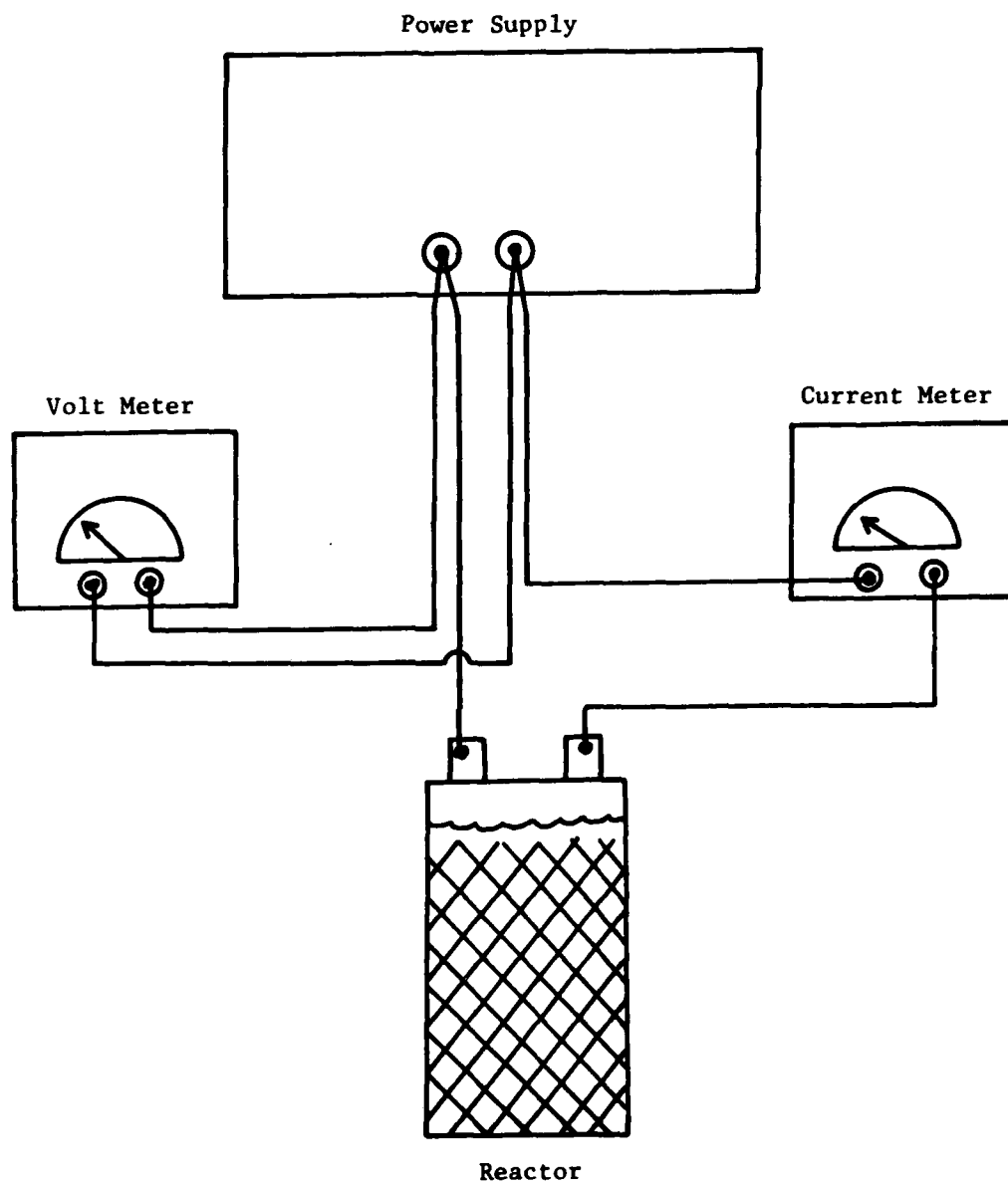


Figure 1. Innova apparatus.

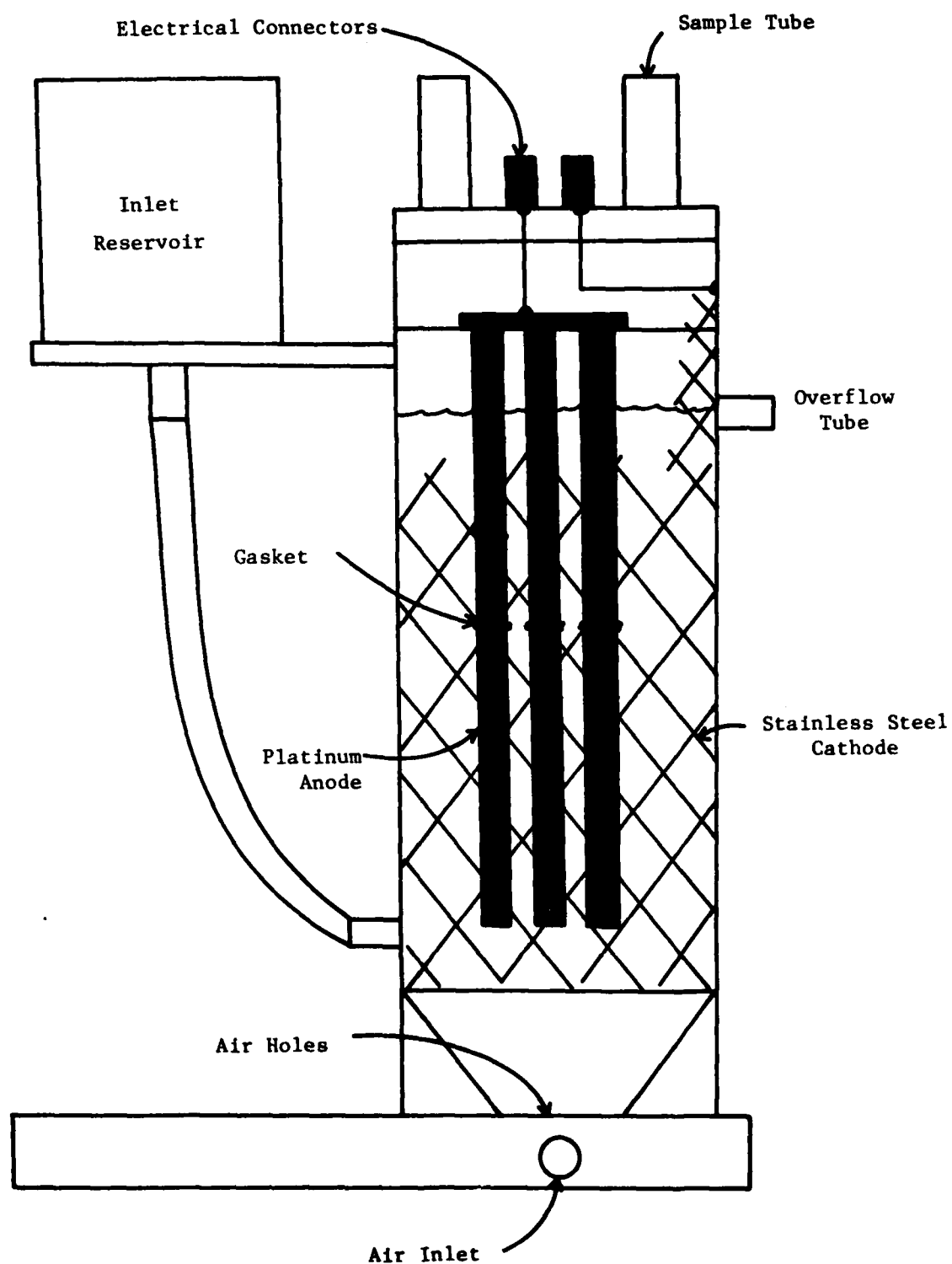
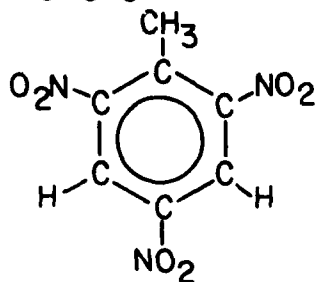
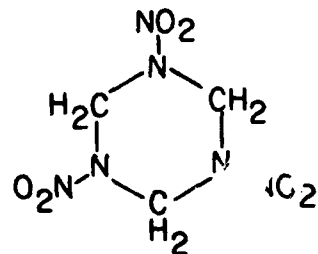


Figure 2. Innova apparatus: reactor details.

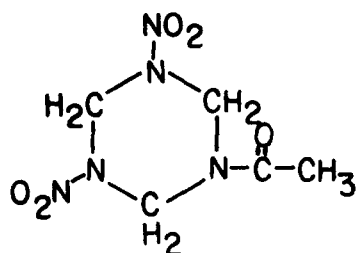
TNT $C_7H_5N_3O_6$
MW 227



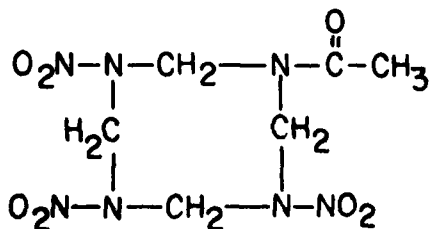
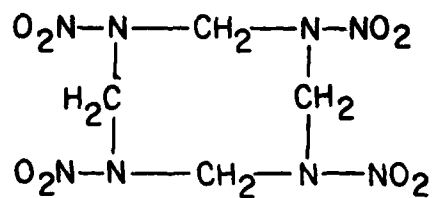
RDX $C_3H_5N_6O_6$
MW 222



TAX $C_5H_9N_5O_5$
MW 219



HMX $C_4H_8N_8O_8$
MW 296



SEX $C_6H_{11}N_7O_7$
MW 293

Figure 3. Structures of test compounds.

EXPERIMENTAL METHODS

The following conditions were used for all experimental runs:

1. An initial working volume of 2.5 L was used.
2. Four hundred and fifty milliliters of dry fiber balls (zeta particles) were used.
3. Sodium sulfate (Na_2SO_4 , 1,000 mg/L) was chosen as a standard for TDS.
4. All runs had a duration of 2 hours.
5. An initial 15-min mixing time was used to establish a random complete mix before the voltage was applied.
6. Samples were taken every 15 min.
7. The amperage was held constant throughout the run at 3A, DC.
8. After each run the fiber balls were dried, washed with distilled water, and redried. After all the virgin fiber particles were used, the washed fiber balls were reused.

During each run the ambient air temperature and the reactor fluid temperature were measured at each sample time. A Presto-Tek model DP-03 conductivity meter was used to measure the conductivity of the makeup water and of the reactor contents at each sample time. The voltage and amperage were also recorded at 15-min intervals.

Each munition was dissolved in distilled deionized water with 1,000 ppm Na_2SO_4 as the only additive. Two 2-hr runs were made on each solution of one munition. A final pair of runs was made with a mixture of all five munition compounds. Each stock solution was prepared the day before the run. After the distilled deionized water was heated to 60°C, the munition and electrolyte were added and stirred overnight. The concentration of munition was estimated by weight added and verified by laboratory analysis.

During each test block three side experiments were performed. The first side experiment tested for thermal degradation of the munition. A portion of the stock solution, usually 1 L, was set aside in a glass beaker and kept between 55-60°C for 2 hr. (The reactor would eventually heat up to approximately 50°C in a 2-hr run.) Initial and final samples were taken. The second experiment tested the adsorption properties of the fiber balls. Approximately 200 mL dry volume of fiber balls were put in 1 L of stock solution in a glass beaker. This mixture was stirred for 2 hr. Once again initial and final samples were taken. The third side experiment tested for the presence of oxides of nitrogen (NO_x) in the off gas from the reactor.

ANALYTICAL METHODS

Munitions removals were profiled over the duration of the run. Two random duplicates, a spiked sample, and a distilled deionized water blank were taken for every run. The initial TNT runs at 1,000 mg/L Na_2SO_4 were analyzed by gas

chromatography. All the other runs were analyzed for munitions by high performance liquid chromatography (HPLC) using a procedure developed at USAMBRDL.⁵ Total carbon, inorganic carbon, organic carbon, free $\text{NO}_x\text{-N}$, total Kjeldahl nitrogen (TKN), and $\text{NH}_3\text{-N}$ were measured at 15-min intervals in the initial TNT work. For the remaining munitions compounds, only an initial and final sample were taken for a carbon and nitrogen balance. The standard operating procedures for each test are as follows:

Total Kjeldahl nitrogen analysis was performed according to EPA Method 351.4⁶ modified as described in Appendix B; nitrate-nitrite nitrogen analysis was carried out according to the automated colorimetric cadmium reduction procedure of EPA Method 353.2;⁶ total organic carbon (TOC) analysis was based on Part 505 of Standard Methods⁷ using the Beckman Model 915 TocamasterTM; and ammonia nitrogen was measured using the Orion Model 95-10 electrode as described in Part 417E of Standard Methods.⁷

Figure 4 shows the apparatus needed to sample the gas for NO_x according to the Saltzman method.⁸ The off gas from the reactor was pulled through the permanganate bubbler and absorbing reagent bubbler by the vacuum pump. The vacuum pump was started just prior to applying the voltage to the reactor and allowed to run through the duration of the run. At the end of the 2-hr run, the absorbing reagent from the bubbler was compared to stock absorbing reagent at 550 nm on a spectrophotometer. The calibration curve is presented in Figure 5. The test for NO_x was done once for each munition compound.

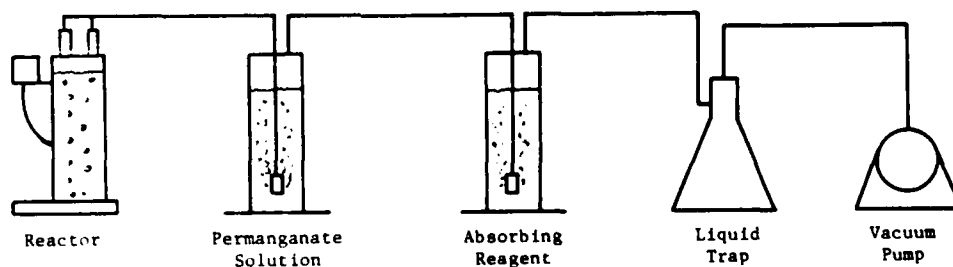


Figure 4. Apparatus for gas analysis by the Saltzman method.

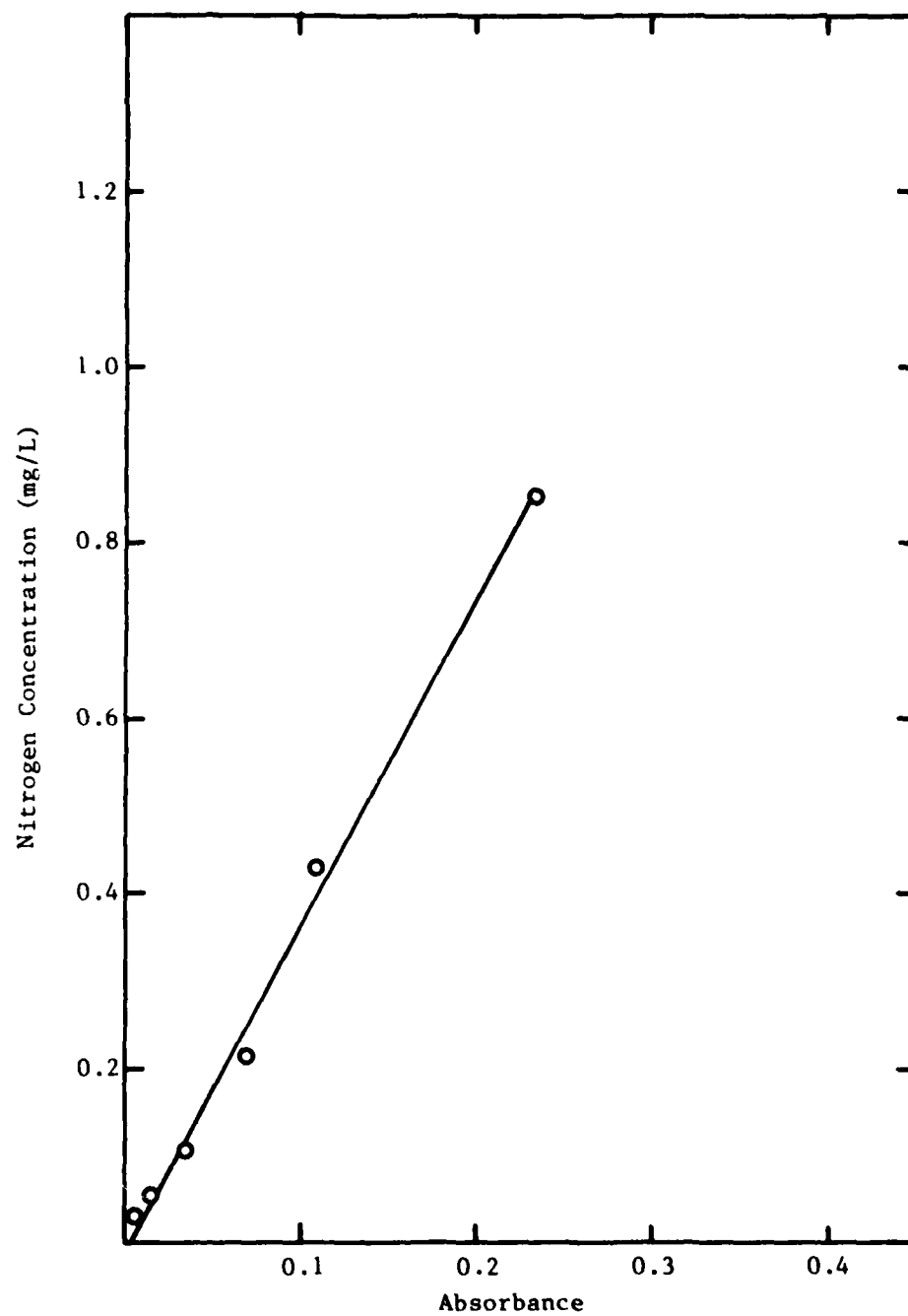


Figure 5. Calibration curve for Saltzman method.

RESULTS AND DISCUSSION

PRELIMINARY WORK

The first experiment was designed to determine the current carrying characteristics of the system. Two and one-half liters of distilled water and 450 mL dry volume of fiber balls were added to the reactor and allowed to mix completely. Sodium sulfate (100 to 1,000 mg/L) was added and allowed to dissolve completely, and the current was then measured at various voltage settings. These data are presented in Table 2 and Figure 6. Only at Na_2SO_4 concentrations above 250 mg/L could stable 3 amp readings be maintained. Because of known TDS wastewater discharge limitations, 1,000 mg/L Na_2SO_4 was chosen as the upper electrolyte concentration limit. During the experiment the water temperature rose only from 24.8°C to 25.1°C.

TABLE 2. EFFECT OF SALT CONCENTRATION ON CONDUCTIVITY
(LEAST SQUARES ANALYSIS)

| Electrolyte Concentration Na_2SO_4 in distilled deionized water (mg/L) | Conductivity, micromhos | Slope | Y Int | Corr. | Temp, °C |
|--|----------------------------|-------|-------|-------|----------|
| 0 | 4 | 125 | 2.33 | 0.987 | 24.8 |
| 101.6 | 230 | 34.1 | 2.01 | 0.996 | 24.7 |
| 246.8 | 530 | 19.3 | 1.94 | 0.997 | 24.8 |
| 496.8 | 900 | 12.7 | 1.24 | 0.997 | 24.9 |
| 746.8 | 1,300 | 9.2 | 1.70 | 0.995 | 25.1 |
| 994.8 | 1,600 | 7.1 | 2.09 | 0.989 | 25.1 |

The second experiment was designed to determine the effect of volume on the current. A 2.5 L solution of 498.7 mg/L Na_2SO_4 was prepared. The reactor was charged with 1 L of solution and 450 mL of dry fiber particles. After a mixing pattern was established, the voltage was turned on. A 3 amp current reading could not be attained, and the 2 amp current reading was unstable; therefore, 1 amp and 26 volts were picked as starting values. The remainder of the stock solution was added in 0.25 L portions. After each addition, the system was allowed to equilibrate before the temperature and amperage readings were taken. The applied voltage remained constant. Amperage/volume data are presented in Figure 7. The system appears to be dependent on volume, which can be related to submerged electrode surface area. (The fiber particle velocities also appeared to vary with volume, but this effect cannot be determined from these data.) This experiment shows that the total volume of samples removed during the 120 minute run should be kept to a minimum, preferably less than 250 mL.

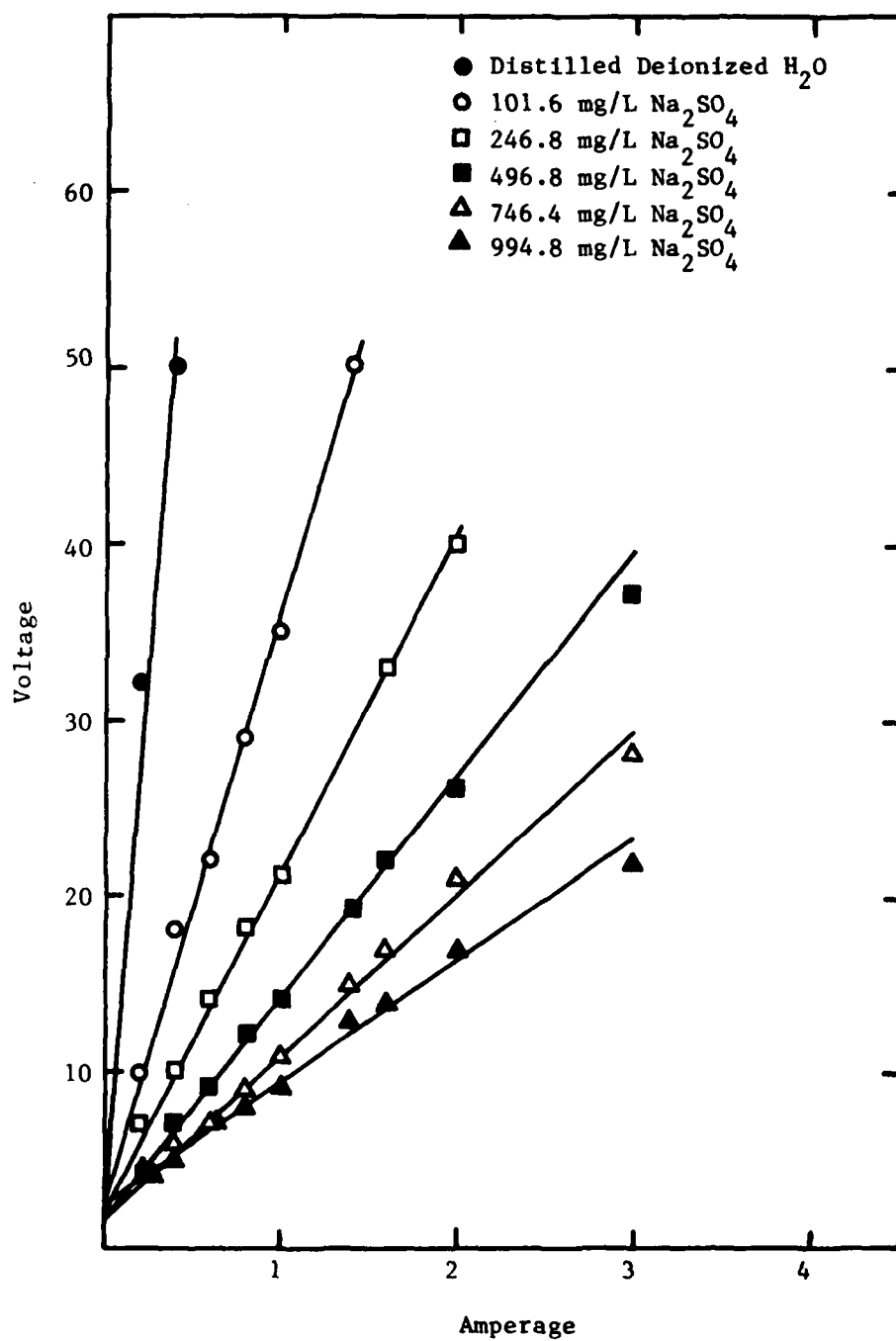


Figure 6. Effect of salt concentration on current.

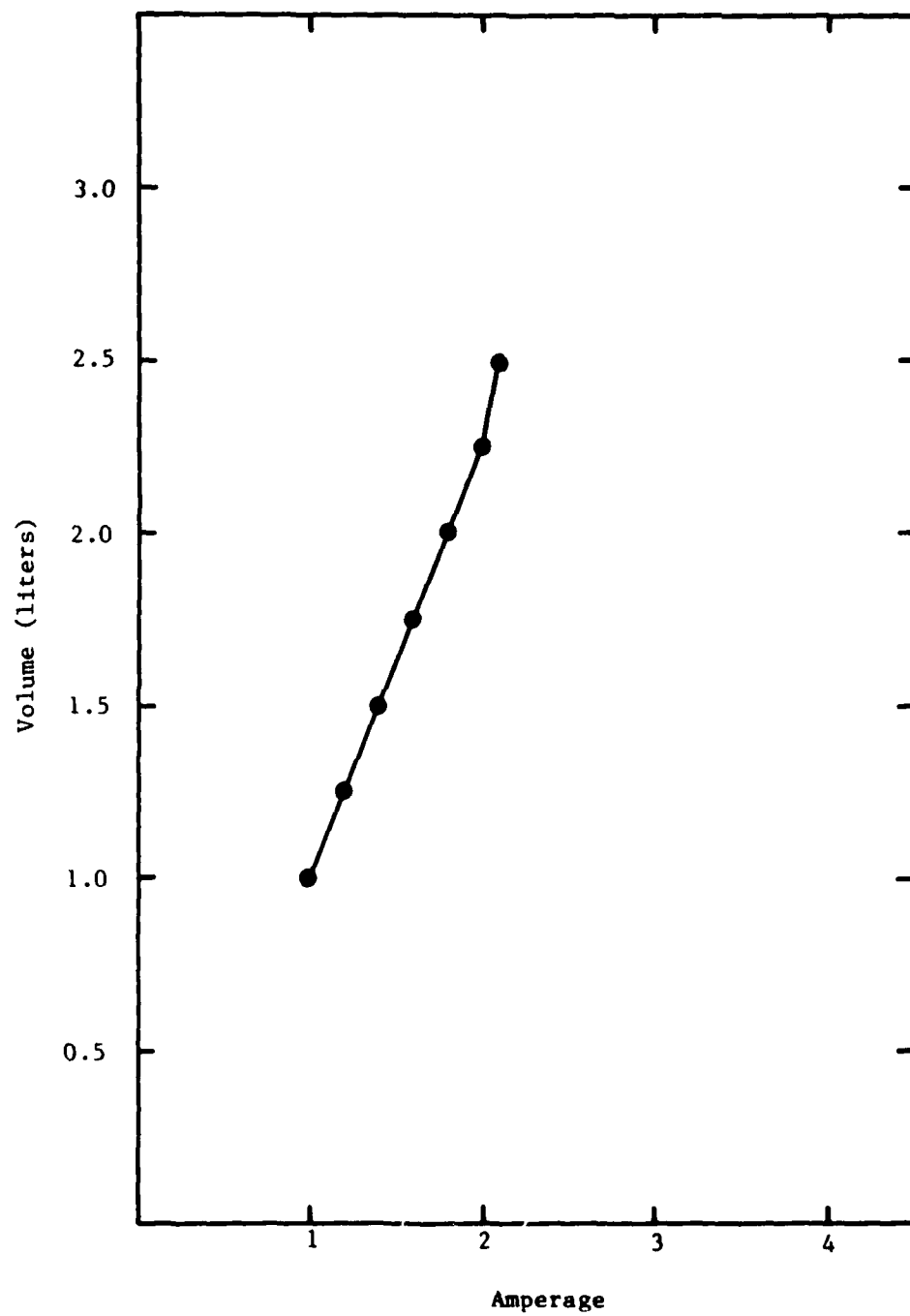


Figure 7. Effect of reactor volume on current.

The third experiment was designed to determine the effect of temperature on amperage. A 2.5 L solution containing 498.7 mg/L Na_2SO_4 was prepared and was added to the reactor along with 450 mL of dry fiber particles. Over a 75-minute time period, the temperature increased by 25.7°C . The amperage-temperature relationship is shown in Figure 8. The relationship is linear. Without making major process and reactor changes in the bench unit, the temperature cannot be controlled.

These experiments show that current characteristics will change over the duration of the run. As samples are taken, a small drop in the current will occur. As the run progresses, the temperature will rise because of the electrical energy input; as the temperature rises, both the amperage and conductance of the system will increase. In order to maintain a constant amperage, the applied voltage must be periodically reduced. Therefore, all 15-minute sample periods do not have the same electrical energy input, which must be taken into account when interpreting the data. Comparisons showing the remaining explosive concentration versus the time the system is exposed to a 3 amp current and the remaining explosive concentration versus the electrical energy in kilowatt hours (KWH) will be presented.

A series of experiments was run on each munition to determine its disappearance by thermal degradation, air stripping at normal flow rates, and adsorption onto the fiber particles. The results of these experiments are presented in Table 3. Air stripping was not done on HMX, SEX, and TAX because each of these compounds has a low volatility, as demonstrated by the thermal degradation experiments. (If any reduction of the munitions had been observed from the thermal degradation work, the air stripping experiments would have been carried out to determine if the loss was through evaporation or through thermal instability.)

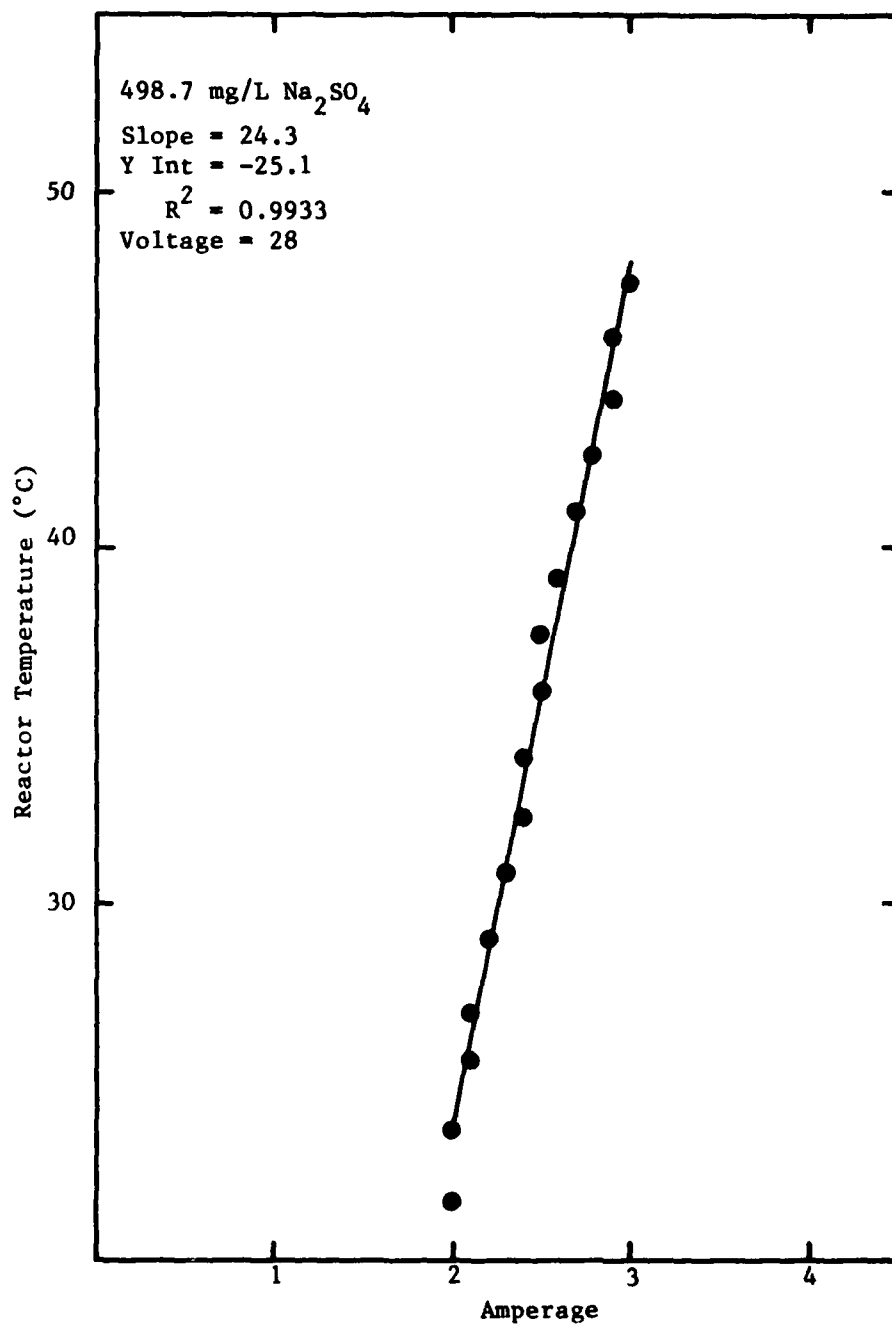


Figure 8. Effect of reactor temperature on current.

TABLE 3. EXPERIMENTAL RESULTS FOR MUNITIONS
REMOVAL BY ADSORPTION AND STRIPPING

| Time, min | Effect of Temperature 50-60°C, mg/L | Effect of Adsorption onto Fiber Particles, mg/L | Effect of Air Stripping, mg/L |
|--------------|--|--|--|
| <u>TNT</u> | | | |
| 0 | 103.0 | 106.8 | 102.7 |
| 120 | 113.7 | 99.8 | 98.9 |
| <u>RDX</u> | | | |
| 0 | 29.84 | 29.93 | 30.09 |
| 120 | 33.23 | 29.47 | 29.10 |
| <u>HMX</u> | | | |
| 0 | 4.61 | 4.47 | -- |
| 120 | 9.65 | 4.20 | -- |
| <u>SEX</u> | | | |
| 0 | 22.85 | 21.83 | -- |
| 120 | 29.31 | 22.19 | -- |
| <u>TAX</u> | | | |
| 0 | 40.22 | 36.29 | -- |
| 120 | 40.78 | 39.09 | -- |

TNT

A total of six runs were made using TNT alone (see Appendix Tables C-1 through C-6). Runs 1, 3, and 4 were made to verify the data reported by Innova. Run 2 was carried out at 1 amp DC in order to facilitate the isolation of intermediates. Runs 5 and 6 were performed at 30 mg/L TNT to serve as direct comparisons to other removal technologies. Figure 9 shows the disappearance of TNT with time. Lack of fit to a linear model (i.e., zero order kinetics) is apparent (except for Run #1, for which only two points were recorded). After transformation of the y, or concentration, variable to the log basis, some curvature remained for data taken after 60 minutes; hence, data >60 min (three points in all from run #4) were discarded. Linear (first-order) models were fitted to the truncated data and tested for equality of slopes using ANCOV (analysis of covariance).⁹ Significant differences in the slopes (Table 4) were detected ($p < 0.01$). Because the slope for Run #2 is most exceptional, Run #2 was omitted and the data were refitted and

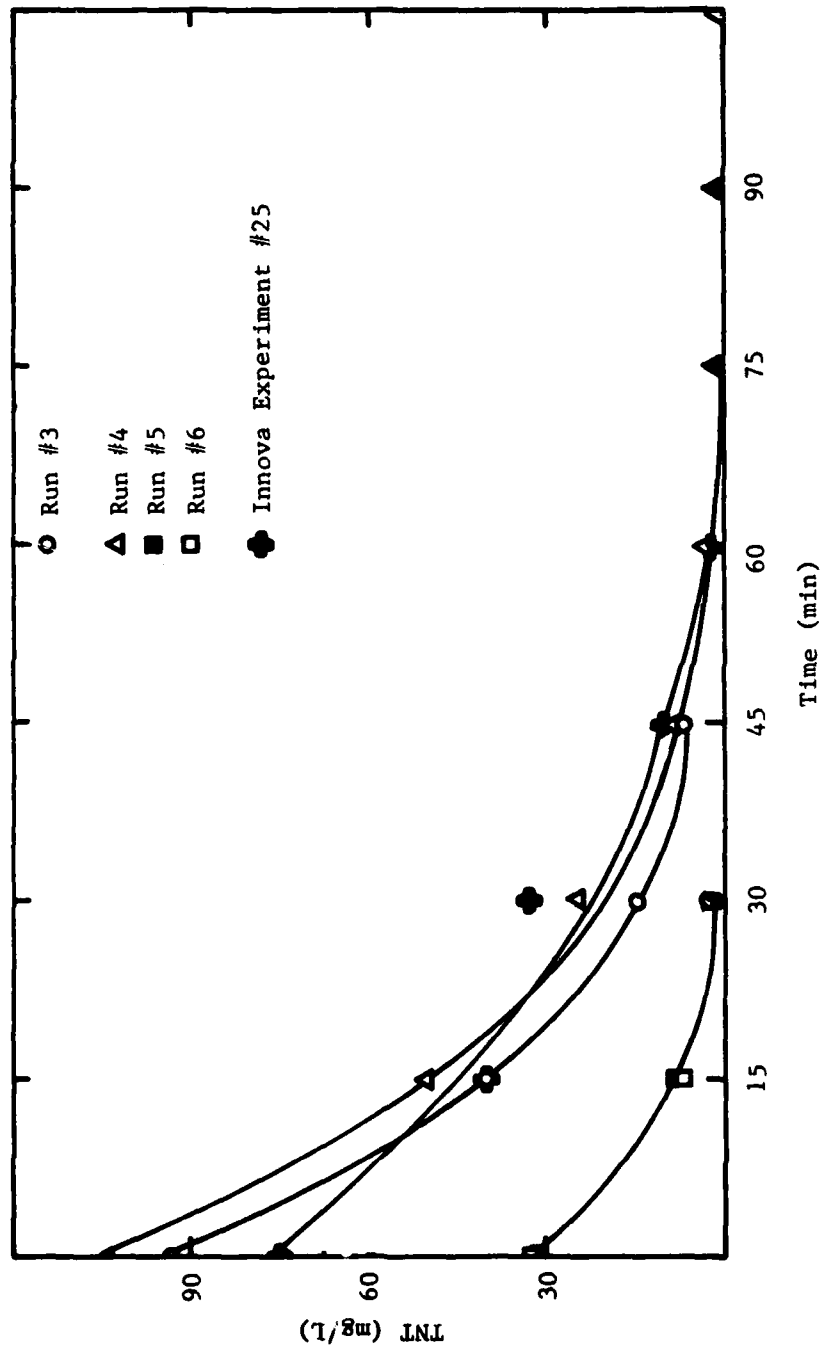


Figure 9. Disappearance of TNT with time.

retested. Significant differences ($p < 0.01$) in the slopes were still detected. ANCOV, using the first-order model and truncated data, defines three distinct groups according to slopes:

| <u>Group</u> | <u>Runs</u> |
|--------------|-------------|
| 1 | 2 |
| 2 | 3,4 |
| 3 | 5,6 |

The low specific rate for Run #2 is probably the result of the low power loading relative to the other runs. The reason for the existence of Groups 2 and 3 is not clear. The only difference in Runs 3 and 4 vs. Runs 5 and 6 is in the initial TNT concentration, which should not affect first-order rate constants. A simple explanation is that accumulation of intermediate products is inhibitory, i.e., the reaction is not truly first order.

TABLE 4. TNT DATA FITTED TO $\ln C/C_0 = Kt + A^a$

| <u>Run</u> | <u>Slope, min^{-1}</u> | <u>S.E. for Slope^b</u> |
|------------|--|-----------------------------------|
| 1 | (-0.099) ^c | - |
| 2 | -0.012 | 0.0021 |
| 3 | -0.060 | 0.0030 |
| 4 | -0.055 | 0.0021 |
| 5 | -0.081 | 0.0046 |
| 6 | -0.098 | 0.0046 |

- a. A, the intercept, is negligible in all cases.
- b. Standard error based on 10 degrees of freedom.
- c. Two points only; not included in ANCOV.

Data provided by Innova² suggest that disappearance of TNT follows first-order kinetics with respect to energy input, E. Since the voltage was altered substantially to limit the current during all runs, first-order plots with respect to time and respect to E are not coincident. TNT degradation data are analyzed with respect to E in Table 5 and plotted in Figure 10. It is noted that major differences in slopes are reduced. Results from the Innova experiments exhibit substantially higher negative slopes for similar initial TNT concentrations. The Innova runs were made using Florida tapwater, perhaps containing oxidizable substances, such as chloride, which could participate in destruction of TNT. (Sodium sulfate would not be expected to participate.) It is further noteworthy that the reactor temperature rose substantially during the course of each reaction (see Appendix Tables C-1 through C-6), but this did not result in enhancement of first order rate constants, whether as a function of time or energy input.

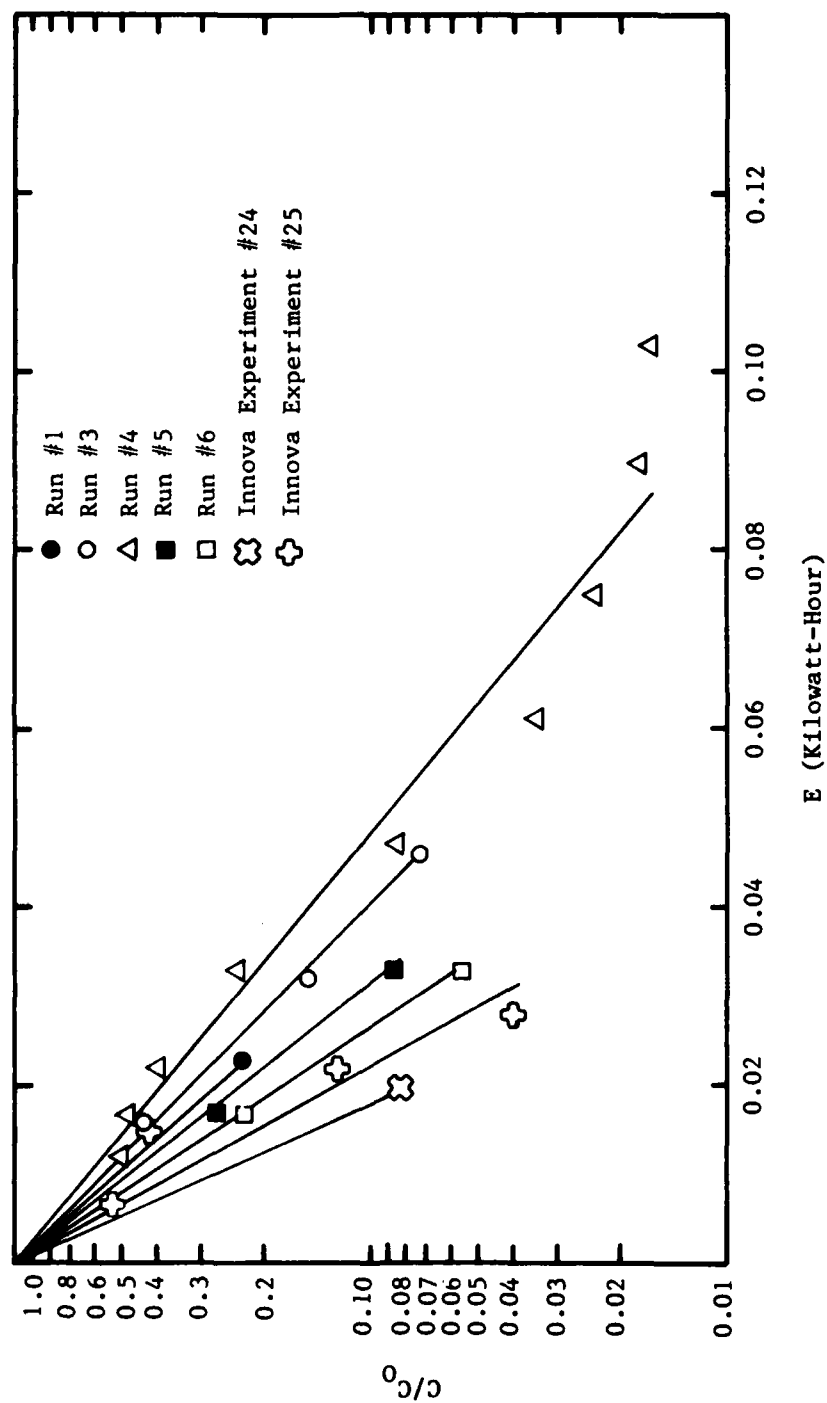


Figure 10. Disappearance of TNT with energy input.

TABLE 5. TNT DATA FITTED TO $\ln C/C_0 = KE + A$

| Run | Slope, ^b KWH^{-1} (Corr.) | C_0 , mg/L |
|------------------------------|--|--------------|
| #1 | -62 ^c | 61.1 |
| #2 | -72 (0.9496) | 7.9 |
| #3 | -58 (0.9986) | 93.5 |
| #4 | -52 (0.9940) | 104.6 |
| #5 | -74 (0.9995) | 31.2 |
| #6 | -87 (0.9999) | 32.7 |
| Innova Exp. #24 ^d | -120 | 65.7 |
| Innova Exp. #25 ^d | -110 | 74.8 |

a. A, the intercept, is essentially zero in all cases.

b. Least squares analysis.

c. Two data points only.

d. Reference 2.

One approach to treatment of the data is to assume that the mechanism and rate of TNT degradation are the same at comparable TNT concentrations, since the driving force for the reaction is the electrical energy input. Then the TNT degradation lines can be overlayed to yield one line by displacing time zero (or energy zero) to the right for the run with a smaller initial concentration. This approach is demonstrated in Figure 11 for concentration vs. time and concentration vs. energy. The correlations appear to be satisfactory, although the Innova data do not fit well.

RDX, HMX, SEX, AND TAX

The other four munitions compounds (all nitramines) were tested under the same conditions of salt concentration and amperage as for TNT. Each compound was reduced to the detection limit or below within 2 hours (see Appendix C). As shown in Table 6, nitramine decomposition follows first order kinetics with respect to time or energy input with fair to good correlation. RDX, SEX, and TAX degrade at essentially the same rate; the rate for HMX appears to be moderately greater, but the initial concentration of HMX was necessarily much smaller, and direct comparison is difficult. Further work is needed to determine precise kinetics and mechanism for nitramine degradation.

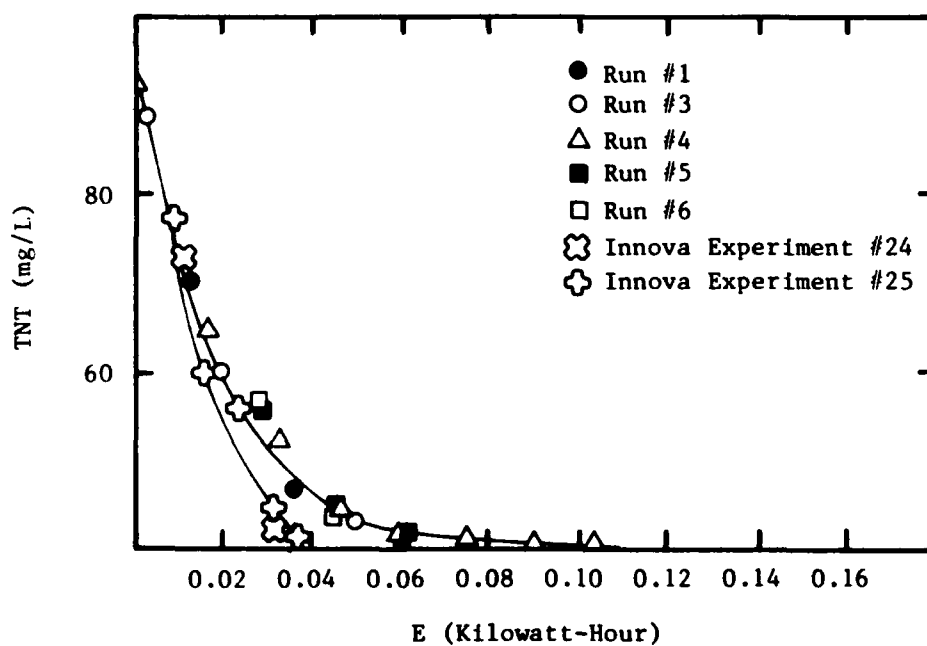
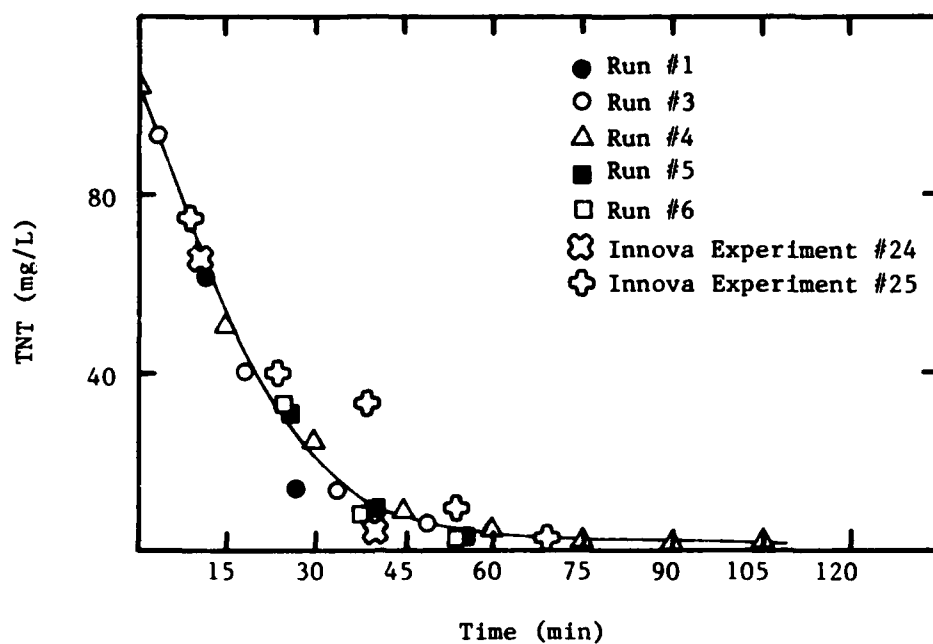


Figure 11. TNT runs (displaced time or energy origins).

TABLE 6. FIRST ORDER RATE CONSTANTS FOR NITRAMINE DECOMPOSITION

| Nitramine | Run | C ₀ , mg/L | -k, min ⁻¹ (Corr.) | -k, KWH ⁻¹ (Corr.) |
|-----------|-----|-----------------------|-------------------------------|-------------------------------|
| RDX | 1 | 25.84 | 0.038 (0.9950) | 48 (0.9862) |
| | 2 | 28.10 | 0.030 (0.9986) | 36 (0.9971) |
| HMX | 1 | 4.73 | 0.047 (0.9764) | 52 (0.9847) |
| | 2 | 4.58 | 0.056 (0.9943) | 63 (0.9963) |
| SEX | 1 | 23.55 | 0.039 (0.9990) | 47 (0.9977) |
| | 2 | 19.65 | 0.037 (0.9980) | 47 (0.9932) |
| TAX | 1 | 35.01 | 0.040 (0.9993) | 47 (0.9954) |
| | 2 | 39.78 | 0.034 (0.9959) | 43 (0.9900) |

MIXTURE RUNS

Replicate runs were made containing a mixture of all five munitions compounds at roughly the same initial concentrations as used with the original single-munition runs (see Appendix C). In each of the two runs only SEX was not removed to below detection limits within 2 hours. First order rate constants for disappearance of munitions from the mixture are presented in Table 7. Comparison with Tables 4, 5, and 6 indicates that degradation of the nitramines is somewhat retarded by the presence of the other munitions, while the rate for TNT is perhaps enhanced. From a practical viewpoint, it is noteworthy that removal of total munitions exceeding 100 mg/L to approximate detection limits from a 2.5 L sample required about 0.1 KWH of energy; under the conditions of these experiments, however, more than half of the energy was wasted in heating the sample.

TABLE 7. FIRST ORDER RATE CONSTANTS FOR MUNITIONS DECOMPOSITION IN MIXTURE

| Munition | Run | C ₀ , mg/L | -k, min ⁻¹ (Corr.) | -k, KWH ⁻¹ (Corr.) |
|----------|-----|-----------------------|-------------------------------|-------------------------------|
| TNT | 1 | 27.87 | 0.100 (0.9966) | 109 (0.9837) |
| | 2 | 33.42 | 0.103 (0.9924) | 108 (0.9999) |
| RDX | 1 | 22.97 | 0.029 (0.9833) | 37 (0.9763) |
| | 2 | 27.56 | 0.033 (0.9851) | 39 (0.9761) |
| HMX | 1 | 3.60 | 0.020 (0.9894) | 26 (0.9881) |
| | 2 | 5.86 | 0.019 (0.9863) | 23 (0.9803) |
| SEX | 1 | 27.74 | 0.021 (0.9524) | 27 (0.9488) |
| | 2 | 21.43 | 0.020 (0.9143) | 22 (0.9361) |
| TAX | 1 | 26.10 | 0.022 (0.9822) | 29 (0.9770) |
| | 2 | 33.94 | 0.030 (0.9820) | 36 (0.9761) |
| SUM | 1 | 108.28 | 0.030 (0.9563) | 38 (0.9550) |
| | 2 | 122.21 | 0.032 (0.9674) | 38 (0.9654) |

CARBON

Table 8 presents the initial and final carbon levels from the reactor as well as calculated organic carbon levels based on GC and HPLC analysis and munition elemental compositions (Table 9). The initial and calculated organic carbon values tend to agree. Problems were encountered with intermediate and final carbon analyses in that fiber particles tend to shed small graphite (carbon) fibers, which stay suspended in solution, foul the TOC analyzer column, and distort the results. (The distilled deionized water had organic carbon levels averaging between 1 and 2 mg/L.) No consistent trends appear concerning inorganic carbon production; organic carbon values do not change or tend to increase over the 2-hour runs (probably due to interference from the graphite). From the carbon data it appears that complete oxidation of carbon is not occurring, and that the munitions are being fragmented to smaller segments, such as oxalic acid for TNT and formic acid for the nitramines.

TABLE 8. CARBON BALANCE

| Experiment | Time | Total Carbon, mg/L | Inorganic Carbon, mg/L | Organic Carbon, mg/L | Calculated Organic Carbon, mg/L |
|----------------|------------------|-----------------------|---------------------------|-------------------------|------------------------------------|
| TNT Run #3 | t ₀ | 42.3 | 0.5 | 41.8 | 34.6 |
| | t ₁₂₀ | 35.4 | 0.8 | 34.6 | |
| TNT Run #4 | t ₀ | 47.5 | 0.5 | 47.0 | 38.7 |
| | t ₁₂₀ | 41.0 | 3.1 | 37.9 | |
| TNT Run #5 | t ₀ | 18.5 | 1.5 | 17.0 | 11.5 |
| | t ₁₂₀ | 28.5 | 7.0 | 21.5 | |
| TNT Run #6 | t ₀ | 17.1 | 1.2 | 15.9 | 12.1 |
| | t ₁₂₀ | 28.5 | 13.0 | 15.5 | |
| RDX Run #1 | t ₀ | 5 | <1 | 5 | 4.2 |
| | t ₁₂₀ | 13 | 1 | 12 | |
| RDX Run #2 | t ₀ | 6 | <1 | 6 | 4.5 |
| | t ₁₂₀ | 12 | <1 | 12 | |
| HMX Run #1 | t ₀ | 3 | <1 | 3 | 0.8 |
| | t ₁₂₀ | 10 | <1 | 10 | |
| HMX Run #2 | t ₀ | 5 | <1 | 5 | 0.7 |
| | t ₁₂₀ | 9 | <1 | 9 | |
| SEX Run #1 | t ₀ | 7 | <1 | 7 | 5.8 |
| | t ₁₂₀ | 13.5 | <1 | 13.5 | |
| SEX Run #2 | t ₀ | 8 | <1 | 8 | 4.9 |
| | t ₁₂₀ | 15 | <1 | 15 | |
| TAX Run #1 | t ₀ | 10 | <1 | 10 | 9.6 |
| | t ₁₂₀ | 16 | <1 | 16 | |
| TAX Run #2 | t ₀ | 12 | <1 | 12 | 10.8 |
| | t ₁₂₀ | 17 | <1 | 17 | |
| Mixture Run #1 | t ₀ | 29 | <1 | 29 | 28.6 |
| | t ₁₂₀ | 27 | <1 | 27 | |
| Mixture Run #2 | t ₀ | 33 | <1 | 33 | 32.6 |
| | t ₁₂₀ | 23 | <1 | 23 | |

TABLE 9. MUNITION ELEMENTAL COMPOSITIONS

| Explosive | Formula | % Carbon | % Nitrogen | % Oxygen | % Hydrogen |
|-----------|-------------------|----------|------------|----------|------------|
| TNT | $C_7N_3O_6H_5$ | 37.0 | 18.5 | 42.3 | 2.2 |
| RDX | $C_3N_6O_6H_6$ | 16.2 | 37.8 | 43.3 | 2.7 |
| HMX | $C_4N_8O_8H_8$ | 16.2 | 37.8 | 43.3 | 2.7 |
| SEX | $C_6N_7O_7H_{11}$ | 24.6 | 33.4 | 38.2 | 3.8 |
| TAX | $C_5N_5O_5H_9$ | 27.4 | 32.0 | 36.5 | 4.1 |

NITROGEN

Nitrogen was also traced as a measure of munitions destruction. Samples were analyzed for soluble NO_x -N, TKN, and NH_3 -N, and the off gas was scrubbed for the presence of NO_x using the Saltzman method. If the munition compounds are totally degraded, the nitrogen should appear as soluble NO_x , gaseous NO_x , gaseous N_2 , or soluble NH_3 . In these experiments an attempt to monitor N_2 was not made. If the munition compounds are only partially degraded, they could form low molecular weight hydrocarbons with NH_2 or NO_2 functional groups, of which only the NH_2 groups would be detected by the TKN analysis. Low molecular weight hydrocarbons with NO_2 functional groups must be picked up by GC or HPLC analysis.

During the initial TNT runs, timed NO_x -N and TKN profiles were measured as shown in Figure 12. The higher the initial TNT concentration, the higher the concentration of NO_x -N and TKN. Table 10 summarizes the nitrogen balance for each run. The available nitrogen is calculated from the initial munition concentration. The percentage of NO_x -N is much higher for TNT than for the other munitions. Overall accountability for nitrogen is highest for TNT, (77-96%). HMX had the second highest recovery of nitrogen. RDX, SEX, and TAX had about the same nitrogen recovery percentages. The two mixture runs demonstrated the lowest accountability for nitrogen, at about 20%.

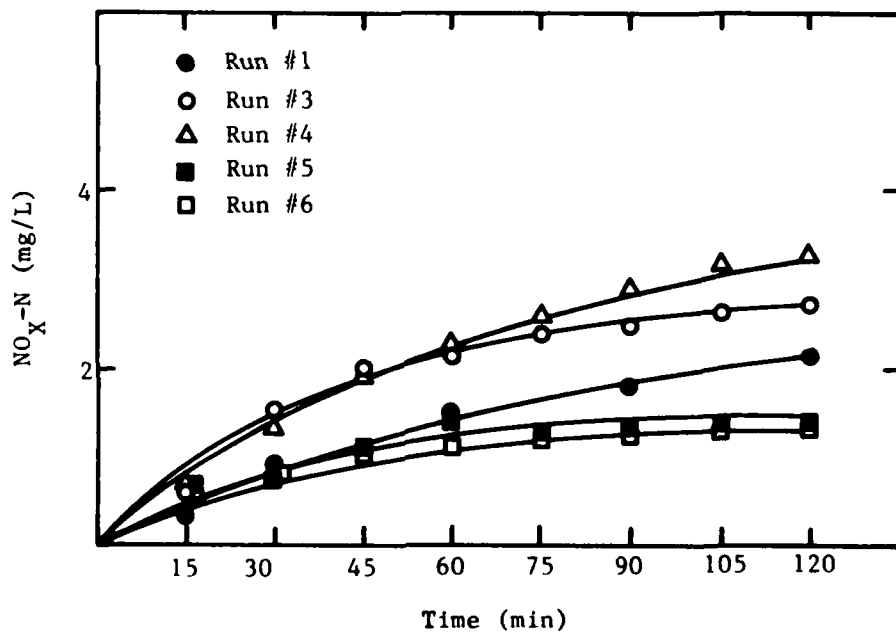
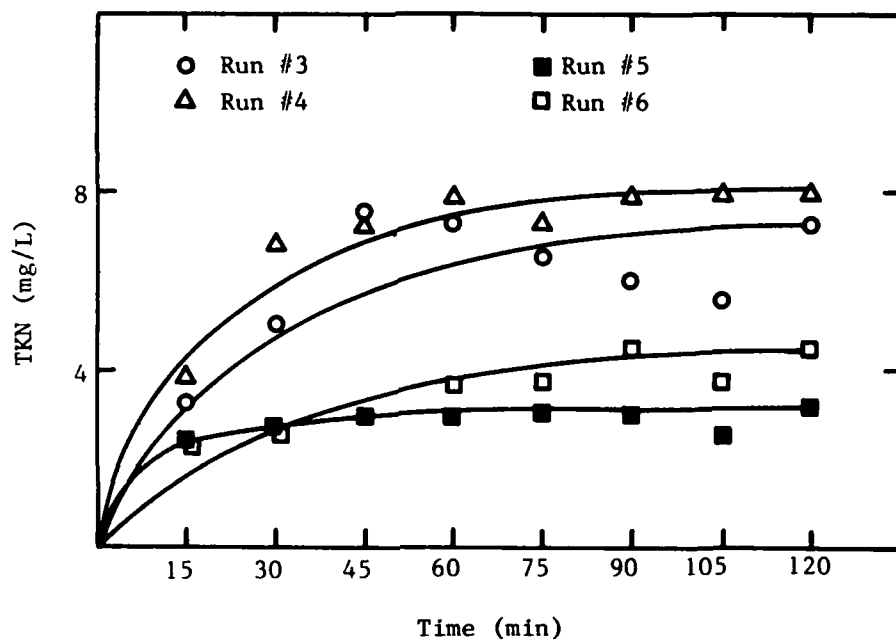


Figure 12. Generation of NO_x and TKN from TNT.

TABLE 10. NITROGEN BALANCE

| Experiment | Calculated Nitrogen from GC/HPLC Analysis | NO ₃ -N Produced, mg/L | TKN Produced, mg/L | NH ₃ -N Produced, mg/L | Total Released, mg/L | % Nitrogen as NO ₃ -N | % Nitrogen Accounted for |
|----------------|---|-----------------------------------|--------------------|-----------------------------------|----------------------|----------------------------------|--------------------------|
| TNT Run #1 | 11.30 | 2.12 | — | — | 2.12 | 18.8 | 18.8 |
| Run #3 | 17.30 | 2.7 | 7.30 | 2.70 | 10.00 | 15.5 | 57.8 |
| Run #4 | 19.35 | 3.2 | 7.85 | 2.18 | 11.05 | 16.5 | 57.1 |
| Run #5 | 5.77 | 1.3 | 3.15 | 1.00 | 4.45 | 22.5 | 77.1 |
| Run #6 | 6.05 | 1.3 | 4.50 | 0.92 | 5.80 | 21.5 | 95.9 |
| RDX Run #1 | 9.87 | 0.85 | 2.52 | 2.73 | 3.37 | 8.6 | 34.1 |
| Run #2 | 10.62 | 0.5 | 2.40 | 2.43 | 2.90 | 4.7 | 27.3 |
| HMX Run #1 | 1.79 | — | 0.93 | 0.64 | 0.93 | — | 52.0 |
| Run #2 | 1.73 | 0.1 | 1.00 | 0.88 | 1.10 | 5.8 | 63.6 |
| SEX Run #1 | 7.87 | 0.1 | 2.90 | 1.50 | 3.00 | 1.3 | 38.1 |
| Run #2 | 6.61 | 0.5 | 2.10 | 1.30 | 2.60 | 7.6 | 39.3 |
| TAX Run #1 | 11.20 | 0.9 | 3.31 | 1.85 | 4.21 | 8.0 | 37.6 |
| Run #2 | 12.66 | 0.2 | 2.40 | 0.87 | 2.60 | 1.6 | 20.5 |
| Mixture Run #1 | | | | | | | |
| TNT | 5.16 | — | — | — | — | — | 23.3 |
| RDX | 8.68 | — | — | — | — | — | — |
| HMX | 1.36 | — | — | — | — | — | — |
| SEX | 9.27 | — | — | — | — | — | — |
| TAX | 8.35 | — | — | — | — | — | — |
| Total | 32.82 | — | 7.64 | 5.05 | 7.64 | — | — |
| Run #2 TNT | 6.18 | — | — | — | — | — | — |
| RDX | 10.42 | — | — | — | — | — | — |
| HMX | 2.22 | — | — | — | — | — | — |
| SEX | 7.16 | — | — | — | — | — | — |
| TAX | 10.86 | — | — | — | — | — | — |
| Total | 36.84 | 0.9 | 6.50 | 3.75 | 7.40 | 2.4 | 20.1 |

Since such a large part of the nitrogen remained unaccounted for, the off gas was checked for the presence of NO_x . Table 11 presents the results of the Saltzman method. As can be seen, the amount of nitrogen leaving the system as NO_x is not significant for any munition.

TABLE 11. GENERATION OF NO_x

| Run | Nitrogen Released in Gas, mg |
|----------------|------------------------------------|
| TNT Run #7 | 0.02 |
| RDX Run #1 | 0.025 |
| HMX Run #1 | -- |
| SEX Run #1 | -- |
| TAX Run #1 | -- |
| Mixture Run #1 | 0.052 |

Two possibilities still exist for completion of a nitrogen balance. Nitrogen gas (N_2) could be generated at the electrodes. The other possibility is the presence of low molecular weight organics having NO_2 as a functional group. As indicated in the carbon analysis, complete oxidation is probably not taking place; therefore, the presence of low molecular weight hydrocarbons containing NO_2 is quite possible.

GAS CHROMATOGRAPHY/MASS SPECTROSCOPY (GC/MS) ANALYSIS

Timed samples were taken from the reactor for analysis by GC/MS in order to track the appearance and disappearance of intermediates and final products. Each sample's soluble phase was extracted with methylene chloride for injection.

The GC/MS verified the disappearance of TNT with time. With the exception of a single sample wherein 2-amino-4,6-dinitrotoluene was identified, no other compounds, and in particular no aromatic products, were detected. (Oxalic acid, a likely TNT degradation product, is highly water soluble and would not appear in the methylene chloride extract.)

The GC/MS analyses of RDX, HMX, SEX, and TAX were similar; these four munitions decomposed in the injection port. The decline of the thermal decomposition products could be traced with time, but no degradation products could be identified. (Formaldehyde and formic acid are possible degradation products but neither of them would be detected using this technique.)

SUMMARY AND CONCLUSIONS

1. The explosive (munition) compounds are individually degraded by the Innova process within a 2-hour time period and can be effectively degraded in a multicomponent system.
2. The explosive compounds are not totally oxidized to CO_2 .
3. Nitrogen is both oxidized and reduced to soluble NO_x and NH_3 ; however, less than 1 percent of the available nitrogen comes off as NO_x gas from RDX and TNT and no NO_x gas was detected during the degradation of HMX, SEX, and TAX.
4. Almost no identifiable degradation products were found by GC/MS analysis after extraction into methylene chloride.
5. Explosive compounds do not adsorb to fiber zeta particles.
6. The highest temperature reached in the process (57°) will not degrade or volatilize the explosive compounds.

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APPENDIX A

INNOVA MANUAL

OXIDATION UNIT INSTRUCTION MANUAL

The Innova oxidation/reduction/coagulation unit consists of specialized particles kept in motion between two electrodes. This manual provides a description of the unit, procedures for start-up, and some trouble-shooting comments. The unit is relatively simple to operate, requiring only a power supply and a source of air for agitation.

Unit Description

The Innova oxidizer is basically a Lucite cylinder with appropriate electrodes. Two openings are located in the top of the unit for sample removal and temperature measurements. Although this unit is usually run in a batch mode, it can be operated for flow testing by using the upper input reservoir and the output tube. The internal base of the unit is conical, with small air holes located in the center.

The air connection is at the base of the unit. The small air pump supplied provides sufficient agitation to keep the particles in motion, providing the air holes are reasonably clear.

Two types of zeta particles have been supplied with the unit. The standard particle is the fiber ball. This particle will sometimes float at the top of the unit and should be evacuated prior to use in the oxidizer. The solid particle is currently under extensive evaluation at Innova and has shown several desirable characteristics under operation in the electric field.

The anodes suspended in the center of the unit are platinum-plated titanium. The cathode around the outside is expanded sheet stainless steel. The red connector on the top of the unit is attached to the anode. The black connector is attached by an alligator clip to the cathode. Please note that whenever the top is removed from the unit the cathode connection must be made first. The top of the unit comes apart in two places allowing for liquid to be added and the cathode connections to be made.

An aluminum anode is supplied. This screws on as the bottom half of an anode, in place of one platinum half, when the unit is used as a coagulator.

The unit has a wide range of normal operation depending on the conductivity of the solution to be tested. Normally the cell operates at 20 volts and 0.5 to 1 amps. Depending on the specific sample, operation anywhere from 5 to 50 volts is not unusual to give a reasonable current. These parameters vary with the conductivity of the solution. The power applied should not exceed 60 watts (watts = volts x amps) so that the temperature does not go above 70°C. Lowering the voltage will lower the watts applied and thereby the operating temperature.

Start-up Procedure

1. Remove the top of the unit, with the attached anodes, by disconnecting the alligator clip on the cathode. Add 450 mL (dry volume) of the particles.
2. Add the liquid (either tap water to be spiked with the sample or the entire sample) to the level of the outflow tube. Replace the top of the unit and reconnect the alligator clip to the expanded sheet stainless steel cathode.
3. Attach the air pump. Allow approximately 5 minutes for the motion of the particles to stabilize. Be sure that the majority of the particles are in motion. If a large number of particles are floating at the top, these particles need to be evacuated before the test can be run.
4. Make the electrical connections.
5. Turn on the power to begin the test. Liquid must be in the unit and air agitation must be on whenever power is applied. (Be sure to disconnect the power before removing the top of the unit as contact between the anodes and the cathode will cause a short.)

Trouble-shooting

1. No current registering on the current meter.
 - A. Check to see that the power supply is operational.
 - B. Re-check the wiring.
 - C. Check to see that the alligator clip from the black connector is securely fastened to the expanded sheet cathode at the top of the unit.
 - D. Check to see that the internal connections to the top red and black connectors are still secure and that the red lead is attached to the anode plate.
 - E. Verify that the current meter is operating accurately.
 - F. Make sure there is sufficient conductivity in the sample solution.
2. Insufficient air agitation.
 - A. Verify that the air pump is operating properly.
 - B. Remove the top of the unit to see if the bottom air holes are plugged. If so, a pin on the end of a rod can be used to remove any particles.
 - C. Verify that no leaks are present in the air tubing line.

3. Erratic current (some fluctuation is normal).

A. Verify that sufficient air is present to keep the particles in motion.

B. Remove any particles floating at the top of the unit, and evacuate and replace them.

4. Unusually slow oxidation rates.

A. Verify that sufficient current is present. A higher conductivity may be necessary, such as by the addition of a low concentration of salt to the water if distilled water is the medium.

B. Reduce the power (voltage) if the temperature is too high.

C. Verify that there is sufficient motion of the particles.

If further questions arise, please contact Innova, Inc. 813-577-3888.

APPENDIX B

DETERMINATION OF TOTAL KJELDAHL NITROGEN BASED ON EPA METHOD 351.4

1. Place 1 glass bead in 100 mL TKN flask.
2. Add 10 mL sample.
3. Add 5 mL TKN digestion solution.
4. Place on TKN digestion apparatus. Heat slowly at first, then gradually increase temperature of digester to maximum. Turn on water, so fumes are drawn out into the water trap.
5. Digest for 1/2 hour after fumes have been obtained on the digester.
6. Cool to room temperature.
7. Dilute to 100 mL and determine $\text{NH}_3\text{-N}$ concentration with the NH_3 probe. Set the NH_3 probe to read 1 with the 0.5 mg/L standard solution, and set on 10 W/5 mg/L $\text{NH}_3\text{-N}$.
8. Multiply the answer obtained from the scale reading by 5 to convert answer to mg/L TKN-N.

APPENDIX C

DATA

TABLE C-1. TNT RESULTS
Run #1 61.1 mg/L TNT and 1,000 mg/L NaHCO₃

| Time min | Ambient Temperature, °C | Reactor Temperature, °C | Conductance, Micromhos | Volts DC | AMPS DC | TNT, mg/L | NO _x -N, mg/L |
|-----------------------------------|-------------------------------|-------------------------------|---------------------------|-------------|------------|--------------|-----------------------------|
| 0 | 20.4 | 22 | 1150 | 33 | 3 | 61.1 | 0 |
| 15 | 20.6 | 33 | 1200 | 30 | 3 | 14.0 | 0.34 |
| 30 | 20.9 | 39 | 1250 | 27 | 3 | | 0.88 |
| 45 | 21.1 | 46 | 1300 | 25 | 3 | | |
| 60 | 21.4 | 50 | 1350 | 23 | 3 | | 1.47 |
| 75 | 21.9 | 53 | 1350 | 22 | 3 | | |
| 90 | 22.0 | 56 | 1350 | 22 | 3 | | 1.79 |
| 105 | 22.4 | 57 | 1350 | 22 | 3 | | |
| 120 | 22.1 | 57 | 1350 | 21 | 3 | | 2.12 |
| Dup 1=t ₁₀₅ | | | | | | | |
| Dup 2=t ₁₂₀ | | | | | | | |
| Spike 50/50 | | | | | | 42.6 | |
| DIDEH ₂ O ^a | | | 3 | | | | |

TABLE C-2. TNT RESULTS
Run #2 7.9 mg/L TNT and 1,000 mg/L Na₂SO₄

| Time min | Ambient Temperature, °C | Reactor Temperature, °C | Conductance Micromhos | Volts DC | Amps DC | TNT, mg/L | NO _x -N, mg/L |
|-----------------------------------|-------------------------------|-------------------------------|--------------------------|-------------|------------|--------------|-----------------------------|
| 0 | 24.0 | 22 | 1700 | 10 | 1 | 7.9 | 0.35 |
| 15 | 23.6 | 27 | 1700 | 10 | 1 | 5.3 | 0.40 |
| 30 | 23.2 | 28 | 1700 | 10 | 1 | 4.6 | 0.40 |
| 45 | 23.2 | 28 | 1700 | 10 | 1 | 3.8 | 0.45 |
| 60 | 23.2 | 28 | 1700 | 10 | 1 | 3.7 | 0.45 |
| 75 | 23.6 | 28 | 1700 | 10 | 1 | | 0.50 |
| 90 | 23.6 | 28 | 1750 | 10 | 1 | | 0.50 |
| 105 | 23.4 | 28 | 1800 | 10 | 1 | | 0.55 |
| 120 | 23.2 | 28 | 1800 | 10 | 1 | | 0.55 |
| Dup 1=t ₄₅ | | | | | | 4.1 | 0.50 |
| Dup 2=t ₁₅ | | | | | | 5.3 | 0.40 |
| Spike 50/50 | | | | | | 5.4 | 0.40 |
| DIDEH ₂ O ^a | | | 3 | | | | 0.20 |

a. DIDEH₂O = distilled, deionized water.

TABLE C-3. TNT RESULTS
Run #3 93.5 mg/L TNT and 1,000 mg/L Na₂SO₄

| Time min | Ambient Temp., °C | Reactor Temp., °C | Conductance Micromhos | Volts DC | Amps DC | TNT mg/L | NO _x -N mg/L | TKN mg/L | NH ₃ -N mg/L |
|-----------------------------------|-------------------------|-------------------------|--------------------------|-------------|------------|-------------|----------------------------|-------------|----------------------------|
| 0 | 20.8 | 24.0 | 1700 | 23 | 3 | 93.5 | 0.025 | 1.5 | 0.5 |
| 15 | 21.8 | 29.4 | 1800 | 21 | 3 | 40.1 | 0.60 | 4.7 | |
| 30 | 22.0 | 36.0 | 1850 | 20 | 3 | 14.3 | 1.5 | 6.5 | |
| 45 | 21.8 | 39.6 | 1900 | 18 | 3 | 6.7 | 1.95 | 9.0 | |
| 60 | 21.8 | 41.8 | 1950 | 17 | 3 | <6.0 | 2.10 | 8.8 | |
| 75 | 21.8 | 44.2 | 1900 | 17 | 3 | | 2.35 | 8.0 | |
| 90 | 21.4 | 46.0 | 1950 | 16 | 3 | | 2.45 | 7.5 | |
| 105 | 21.2 | 48.6 | 1000 | 16 | 3 | | 2.60 | 7.0 | |
| 120 | 21.8 | 49.6 | 2000 | 16 | 3 | | 2.70 | 8.8 | 3.2 |
| Dup1=t ₆₀ | | | | | | <6.0 | 2.20 | 8.0 | |
| Dup2=t ₇₅ | | | | | | <6.0 | 2.45 | 8.0 | |
| Spike=50/50 | | | | | | 47.5 | 1.40 | 5.0 | |
| DIDEH ₂ O ^a | | | 3 | | | <6.0 | 0.015 | 0.7 | |

TABLE C-4. TNT RESULTS
Run #4 104.6 mg/L TNT and 1,000 mg/L Na₂SO₄

| Time min | Ambient Temp., °C | Reactor Temp., °C | Conductance Micromhos | Volts DC | Amps DC | TNT mg/L | NO _x -N mg/L | TKN mg/L | NH ₃ -N mg/L |
|-----------------------------------|-------------------------|-------------------------|--------------------------|-------------|------------|-------------|----------------------------|-------------|----------------------------|
| 0 | 22.6 | 26 | 1500 | 23 | 3 | 104.6 | 0.02 | 1.65 | <0.5 |
| 15 | 22.4 | 31 | 1700 | 22 | 3 | 50.9 | 0.65 | 5.5 | |
| 30 | 22.4 | 36 | 1800 | 20 | 3 | 25.0 | 1.35 | 8.5 | |
| 45 | 22.6 | 42 | 1950 | 19 | 3 | 8.8 | 1.95 | 9.0 | |
| 60 | 22.6 | 44 | 1950 | 19 | 3 | 3.7 | 2.30 | 9.5 | |
| 75 | 22.8 | 47 | 1900 | 19 | 3 | 2.5 | 2.60 | 9.0 | |
| 90 | 22.2 | 50 | 2000 | 18 | 3 | 1.9 | 2.90 | 9.5 | |
| 105 | 22.4 | 52 | 1900 | 18 | 3 | 1.8 | 3.15 | 9.5 | |
| 120 | 22.2 | 54 | 1900 | 18 | 3 | | 3.25 | 9.5 | 2.18 |
| Dup1=t ₃₀ | | | | | | 23.6 | 1.35 | 7.5 | |
| Dup2=t ₄₅ | | | | | | 7.3 | 1.95 | 8.5 | |
| Spike 50/50 | | | | | | 67.0 | 1.15 | — | |
| DIDEH ₂ O ^a | | | 4 | | | | 0.005 | 0.65 | |

a. DIDEH₂O = distilled, deionized water.

TABLE C-5. TNT RESULTS
Run #5 31.2 mg/L TNT and 1,300 mg/L Na₂SO₄

| Time min | Ambient Temp., °C | Reactor Temp., °C | Conductance Micromhos | Volts DC | Amps DC | TNT mg/L | NO _x -N mg/L | TKN mg/L | NH ₃ -N mg/L |
|-----------------------------------|-------------------------|-------------------------|--------------------------|-------------|------------|-------------|----------------------------|-------------|----------------------------|
| 0 | 23.0 | 26.4 | 1600 | 24 | 3 | 31.2 | 0.055 | 0.95 | 0.25 |
| 15 | 22.8 | 31.8 | 1750 | 22 | 3 | 8.5 | 0.75 | 3.4 | |
| 30 | 22.9 | 37.0 | 1850 | 20 | 3 | 2.7 | 0.78 | 3.7 | |
| 45 | 22.4 | 41.6 | 1900 | 19 | 3 | | 1.15 | 3.9 | |
| 60 | 22.5 | 45.1 | 1900 | 19 | 3 | | 1.45 | 3.9 | |
| 75 | 22.6 | 47.8 | 2000 | 19 | 3 | | 1.30 | 4.0 | |
| 90 | 22.7 | 50.0 | 1950 | 18 | 3 | | 1.35 | 4.0 | |
| 105 | 22.4 | 54.0 | 1950 | 18 | 3 | | 1.40 | 3.5 | |
| 120 | 22.8 | 55.0 | 2000 | 18 | 3 | | 1.40 | 4.1 | 1.25 |
| Dup1=t ₁₅ | | | | | | 7.8 | 0.65 | 2.6 | |
| Dup2=t ₆₀ | | | | | | | 1.25 | 3.3 | |
| Spike 50/50 | | | | | | 14.6 | 0.15 | -- | |
| DIDEH ₂ O ^a | | | | | | | 0.025 | <0.5 | |

TABLE C-6. TNT RESULTS
Run #6 32.7 mg/L TNT and 1,000 mg/L Na₂SO₄

| Time min | Ambient Temp., °C | Reactor Temp., °C | Conductance Micromhos | Volts DC | Amps DC | TNT mg/L | NO _x -N mg/L | TKN mg/L | NH ₃ -N mg/L |
|-----------------------------------|-------------------------|-------------------------|--------------------------|-------------|------------|-------------|----------------------------|-------------|----------------------------|
| 0 | 23.0 | 23.9 | 1700 | 24 | 3 | 32.7 | 0.04 | 9.0 | 0.23 |
| 15 | 22.5 | 29.2 | 1750 | 22 | 3 | 7.4 | 0.7 | 3.4 | |
| 30 | 22.8 | 35.4 | 1800 | 21 | 3 | 1.8 | 0.9 | 3.65 | |
| 45 | 22.8 | 40.2 | 1800 | 20 | 3 | | 1.1 | -- | |
| 60 | 22.9 | 44.8 | 1850 | 20 | 3 | | 1.15 | 4.6 | |
| 75 | 22.7 | 47.2 | 1800 | 19 | 3 | | 1.25 | 4.7 | |
| 90 | 22.9 | 49.7 | 1800 | 19 | 3 | | 1.35 | 5.5 | |
| 105 | 22.6 | 51.8 | 1900 | 19 | 3 | | 1.40 | 4.7 | |
| 120 | 22.5 | 55.0 | 1950 | 18 | 3 | | 1.35 | 5.5 | 1.15 |
| Dup1=t ₄₅ | | | | | | | 1.1 | 5.0 | |
| Dup2=t ₁₅ | | | | | | 8.9 | 0.7 | 3.9 | |
| Spike 50/50 | | | | | | | 0.75 | 3.3 | |
| DIDEH ₂ O ^a | | | | | | | 0.025 | 0.55 | |

a. DIDEH₂O = distilled, deionized water.

TABLE C-7. RDX RESULTS
Run #1 26.11 mg/L RDX and 1,000 mg/L Na₂SO₄

| Time min | Ambient Temp., °C | Reactor Temp., °C | Conductance Micromhos | Volts DC | Amps DC | RDX mg/L | NO _x -N mg/L | TKN mg/L | NH ₃ -N mg/L |
|-----------------------------------|-------------------------|-------------------------|--------------------------|-------------|------------|-------------|----------------------------|-------------|----------------------------|
| 0 | 23.4 | 24 | 1550 | 22 | 3 | 26.11 | 1.65 | 6.83 | 8.09 |
| 15 | 23.4 | 30 | 1750 | 20 | 3 | 15.72 | | | |
| 30 | 23.3 | 32.8 | 1700 | 19 | 3 | 11.15 | | | |
| 45 | 23.0 | 36.6 | 1800 | 19 | 3 | 6.31 | | | |
| 60 | 23.0 | 38.0 | 1850 | 17 | 3 | 3.38 | | | |
| 75 | 23.0 | 40.6 | 1850 | 17 | 3 | 2.22 | | | |
| 90 | 22.9 | 42.2 | 1900 | 17 | 3 | 1.15 | | | |
| 105 | 22.8 | 44.2 | 1950 | 16 | 3 | 0.46 | | | |
| 120 | 22.8 | 44.6 | 1950 | 16 | 3 | 0.29 | 2.5 | 3.35 | 2.8 |
| Dup1=t ₀ | | | | | | 25.58 | | | |
| Dup2=t ₁₅ | | | | | | 16.20 | | | |
| Spike 50/50 | | | | | | 7.65 | | | |
| DIDEH ₂ O ^a | | | 4 | | | <0.25 | | | |
| Detection limit | | | | | | 0.25 | | | |

TABLE C-8. RDX RESULTS
RUN #2 28.10 MG/L RDX AND 1,000 MG/L Na₂SO₄

| Time min | Ambient Temp., °C | Reactor Temp., °C | Conductance Micromhos | Volts DC | Amps DC | RDX mg/L | NO _x -N mg/L | TKN mg/L | NH ₃ -N mg/L |
|-----------------------------------|-------------------------|-------------------------|--------------------------|-------------|------------|-------------|----------------------------|-------------|----------------------------|
| 0 | 22.9 | 25.2 | 1650 | 22 | 3 | 28.10 | 1.9 | 1.2 | 0.07 |
| 15 | 22.2 | 29.9 | 1700 | 20 | 3 | 20.00 | | | |
| 30 | 22.3 | 34.4 | 1800 | 19 | 3 | 13.55 | | | |
| 45 | 22.5 | 39.6 | 1800 | 18 | 3 | 7.96 | | | |
| 60 | 22.1 | 43.0 | 1800 | 17 | 3 | 4.72 | | | |
| 75 | 22.2 | 45.0 | 1850 | 17 | 3 | 3.24 | | | |
| 90 | 22.5 | 46.8 | 1850 | 17 | 3 | 2.25 | | | |
| 105 | 21.8 | 48.6 | 1900 | 17 | 3 | 1.23 | | | |
| 120 | 22.5 | 50.0 | 1900 | 17 | 3 | 0.78 | 2.4 | 3.6 | 2.5 |
| Dup1=t ₁₅ | | | | | | 20.13 | | | |
| Dup2=t ₃₀ | | | | | | 13.53 | | | |
| Spike 50/50 | | | | | | 24.0 | | | |
| DIDEH ₂ O ^a | | | 4 | | | <0.3 | | | |
| Detection limit | | | | | | 0.3 | | | |

a. DIDEH₂O = distilled, deionized water.

TABLE C-9. HMX RESULTS
Run #1 4.73 mg/L HMX and 1,000 mg/L Na₂SO₄

| Time min | Ambient Temp., °C | Reactor Temp., °C | Conductance Micromhos | Volts DC | Amps DC | HMX mg/L | NO ₃ -N mg/L | TKN mg/L | NH ₃ -N mg/L |
|-----------------------------------|-------------------------|-------------------------|--------------------------|-------------|------------|-------------|----------------------------|-------------|----------------------------|
| 0 | 23.3 | 24.0 | 1500 | 22 | 3 | 4.73 | 0.8 | 0.50 | 0.06 |
| 15 | 22.2 | 30.8 | 1600 | 21 | 3 | 1.94 | | | |
| 30 | 22.5 | 34.4 | 1650 | 19 | 3 | 0.77 | | | |
| 45 | 23.0 | 37.2 | 1700 | 19 | 3 | 0.37 | | | |
| 60 | 22.6 | 39.0 | 1750 | 18 | 3 | 0.32 | | | |
| 75 | 22.7 | 41.2 | 1800 | 18 | 3 | <0.31 | | | |
| 90 | 22.6 | 42.0 | 1900 | 18 | 3 | | | | |
| 105 | 22.7 | 45.4 | 1900 | 18 | 3 | | | | |
| 120 | 22.7 | 43.8 | 1900 | 18 | 3 | | 0.75 | 1.43 | 0.70 |
| Dupl=t ₁₅ | | | | | | 1.91 | | | |
| Dup2=t ₄₅ | | | | | | 0.37 | | | |
| Spike 50/50 | | | | | | 2.46 | | | |
| DIDEH ₂ O ^a | | | 5 | | | <0.31 | | | |
| Detection limit | | | | | | 0.31 | | | |

TABLE C-10. HMX RESULTS
Run #2 4.58 mg/L HMX and 1,000 mg/L Na₂SO₄

| Time min | Ambient Temp., °C | Reactor Temp., °C | Conductance Micromhos | Volts DC | Amps DC | HMX mg/L | NO ₃ -N mg/L | TKN mg/L | NH ₃ -N mg/L |
|-----------------------------------|-------------------------|-------------------------|--------------------------|-------------|------------|-------------|----------------------------|-------------|----------------------------|
| 0 | 23.4 | 25.8 | 1550 | 22 | 3 | 4.58 | 0.6 | 0.55 | 0.07 |
| 15 | 22.8 | 31.0 | 1550 | 21 | 3 | 2.21 | | | |
| 30 | 23.0 | 36.8 | 1550 | 20 | 3 | 0.74 | | | |
| 45 | 22.8 | 41.8 | 1650 | 19 | 3 | 0.30 | | | |
| 60 | 22.7 | 44.0 | 1650 | 18 | 3 | 0.18 | | | |
| 75 | 23.0 | 47.2 | 1700 | 17 | 3 | <0.17 | | | |
| 90 | 22.8 | 48.8 | 1700 | 17 | 3 | | | | |
| 105 | 23.3 | 50.2 | 1700 | 17 | 3 | | | | |
| 120 | 22.8 | 51.6 | 1700 | 17 | 3 | | 0.7 | 1.55 | 0.95 |
| Dupl=t ₃₀ | | | | | | 0.74 | | | |
| Dupl=t ₁₅ | | | | | | 2.10 | | | |
| Spike 50/50 | | | | | | 0.36 | | | |
| DIDEH ₂ O ^a | | | 4 | | | <0.17 | | | |
| Detection limit | | | | | | 0.17 | | | |

a. DIDEH₂O = distilled, deionized water.

TABLE C-11. SEX RESULTS
Run #1 23.55 mg/L SEX and 1,000 mg/L Na₂SO₄

| Time min | Ambient Temp., °C | Reactor Temp., °C | Conductance Micromhos | Volts DC | Amps DC | SEX mg/L | NO _x -N mg/L | TKN mg/L | NH ₃ -N mg/L |
|-----------------------------------|-------------------------|-------------------------|--------------------------|-------------|------------|-------------|----------------------------|-------------|----------------------------|
| 0 | 24.7 | 27.8 | 1500 | 22 | 3 | 23.55 | 2.25 | 1.6 | 0.2 |
| 15 | 23.4 | 30.4 | 1500 | 20 | 3 | 13.02 | | | |
| 30 | 23.3 | 32.8 | 1500 | 19 | 3 | 8.16 | | | |
| 45 | 23.2 | 37.0 | 1550 | 18 | 3 | 4.16 | | | |
| 60 | 22.7 | 38.2 | 1600 | 18 | 3 | 2.18 | | | |
| 75 | 22.7 | 39.2 | 1600 | 17 | 3 | 1.26 | | | |
| 90 | 22.8 | 41.8 | 1600 | 17 | 3 | 0.66 | | | |
| 105 | 23.0 | 42.6 | 1600 | 17 | 3 | 0.43 | | | |
| 120 | 23.2 | 43.8 | 1650 | 16 | 3 | <0.293 | 2.35 | 4.5 | 1.7 |
| Dup1=t ₃₀ | | | | | | 8.56 | | | |
| Dup2=t ₁₅ | | | | | | 13.51 | | | |
| Spike 50/50 | | | | | | 4.47 | | | |
| DIDEH ₂ O ^a | | | 6 | | | <0.293 | | | |
| Detection limit | | | | | | 0.293 | | | |

TABLE C-12. SEX RESULTS
Run #2 19.80 mg/L SEX and 1,000 mg/L Na₂SO₄

| Time min | Ambient Temp., °C | Reactor Temp., °C | Conductance Micromhos | Volts DC | Amps DC | SEX mg/L | NO _x -N mg/L | TKN mg/L | NH ₃ -N mg/L |
|-----------------------------------|-------------------------|-------------------------|--------------------------|-------------|------------|-------------|----------------------------|-------------|----------------------------|
| 0 | 24.6 | 27.6 | 1500 | 22 | 3 | 19.80 | 1.8 | 1.4 | 0.2 |
| 15 | 23.6 | 32.0 | 1500 | 21 | 3 | 12.70 | | | |
| 30 | 23.8 | 37.0 | 1500 | 20 | 3 | 7.74 | | | |
| 45 | 23.2 | 41.6 | 1500 | 18 | 3 | 4.25 | | | |
| 60 | 23.0 | 44.4 | 1600 | 18 | 3 | 2.51 | | | |
| 75 | 22.9 | 46.8 | 1600 | 17 | 3 | 1.30 | | | |
| 90 | 22.9 | 48.6 | 1600 | 17 | 3 | 0.74 | | | |
| 105 | 22.8 | 50.2 | 1650 | 17 | 3 | 0.38 | | | |
| 120 | 22.7 | 51.4 | 1650 | 17 | 3 | 0.30 | 2.3 | 3.5 | 1.5 |
| Dup1=t ₀ | | | | | | 19.50 | | | |
| Dup2=t ₁₅ | | | | | | 12.40 | | | |
| Spike 50/50 | | | | | | 6.50 | | | |
| DIDEH ₂ O ^a | | | 6 | | | <0.293 | | | |
| Detection limit | | | | | | 0.293 | | | |

a. DIDEH₂O = distilled, deionized water.

TABLE C-13. TAX RESULTS
Run #1 35.01 mg/L TAX and 1,000 mg/L Na₂SO₄

| Time min | Ambient Temp., °C | Reactor Temp., °C | Conductance Micromhos | Volts DC | Amps DC | TAX mg/L | NO _x -N mg/L | TKN mg/L | NH ₃ -N mg/L |
|-----------------------------------|-------------------------|-------------------------|--------------------------|-------------|------------|-------------|----------------------------|-------------|----------------------------|
| 0 | 23.8 | 26.6 | 1300 | 22 | 3 | 35.01 | 0.1 | 0.25 | 0.45 |
| 15 | 23.3 | 29.6 | 1400 | 21 | 3 | 18.93 | | | |
| 30 | 23.0 | 33.6 | 1450 | 20 | 3 | 11.35 | | | |
| 45 | 22.9 | 37.8 | 1500 | 19 | 3 | 6.18 | | | |
| 60 | 22.7 | 39.6 | 1500 | 18 | 3 | 3.40 | | | |
| 75 | 22.7 | 41.6 | 1550 | 18 | 3 | 1.89 | | | |
| 90 | 22.8 | 43.4 | 1550 | 17 | 3 | — | | | |
| 105 | 22.7 | 45.4 | 1550 | 17 | 3 | 0.5 | | | |
| 120 | 22.8 | 46.2 | 1600 | 17 | 3 | <0.5 | 3.0 | 5.56 | 2.3 |
| Dupl=t ₁₅ | | | | | | 18.63 | | | |
| Dup2=t ₃₀ | | | | | | 11.53 | | | |
| Spike=50/50 | | | | | | 10.09 | | | |
| DIDEH ₂ O ^a | | | 4 | | | <0.5 | | | |
| Detection limit | | | | | | 0.5 | | | |

TABLE C-14. TAX RESULTS
Run #2 39.57 mg/L TAX and 1,000 mg/L Na₂SO₄

| Time min | Ambient Temp., °C | Reactor Temp., °C | Conductance Micromhos | Volts DC | Amps DC | TAX mg/L | NO _x -N mg/L | TKN mg/L | NH ₃ -N mg/L |
|-----------------------------------|-------------------------|-------------------------|--------------------------|-------------|------------|-------------|----------------------------|-------------|----------------------------|
| 0 | 24.3 | 29.2 | 1400 | 21 | 3 | 39.57 | 2.9 | 2.1 | 0.53 |
| 15 | 24.3 | 34.0 | 1450 | 20 | 3 | 23.95 | | | |
| 30 | 23.8 | 38.0 | 1500 | 20 | 3 | 15.68 | | | |
| 45 | 23.8 | 41.8 | 1550 | 18 | 3 | 9.43 | | | |
| 60 | 23.6 | 45.0 | 1600 | 17 | 3 | 6.33 | | | |
| 75 | 23.6 | 46.8 | 1600 | 17 | 3 | 3.57 | | | |
| 90 | 23.4 | 48.6 | 1600 | 17 | 3 | 2.31 | | | |
| 105 | 23.5 | 50.2 | 1600 | 16 | 3 | 1.27 | | | |
| 120 | 23.5 | 51.2 | 1650 | 16 | 3 | 0.55 | 3.1 | 4.5 | 1.4 |
| Dupl=t ₀ | | | | | | 40.00 | | | |
| Dup2=t ₃₀ | | | | | | 16.24 | | | |
| Spike 50/50 | | | | | | 19.50 | | | |
| DIDEH ₂ O ^a | | | 5 | | | <0.5 | | | |
| Detection Limit | | | | | | 0.5 | | | |

a. DIDEH₂O = distilled, deionized water.

TABLE C-15. MIXTURE RUN #1 REACTOR CONDITIONS (1,000 mg/L Na₂SO₄)

| Time min | Ambient Temperature, °C | Reactor Temperature, °C | Conductance, Micromhos | Volts DC | Amps DC |
|-----------------------------------|-------------------------------|-------------------------------|---------------------------|-------------|------------|
| 0 | 23.9 | 27.0 | 1450 | 22 | 3 |
| 15 | 22.9 | 29.8 | 1550 | 21 | 3 |
| 30 | 22.7 | 34.4 | 1900 | 18 | 3 |
| 45 | 22.5 | 35.6 | 2050 | 17 | 3 |
| 60 | 22.5 | 39.2 | 2100 | 17 | 3 |
| 75 | 22.5 | 42.6 | 2100 | 16 | 3 |
| 90 | 22.4 | 43.8 | 2100 | 16 | 3 |
| 105 | 22.4 | 47.0 | 2100 | 16 | 3 |
| 120 | 22.1 | 48.2 | 2000 | 16 | 3 |
| DIDEH ₂ O ^a | -- | | | | |

TABLE C-16. MIXTURE RUN #1 RESULTS EXPLOSIVES REMOVAL

| Time min | TNT mg/L | RDX mg/L | HMX mg/L | SEX mg/L | TAX mg/L | Σ of all Explosives mg/L | NO _x -N mg/L | TKN mg/L | NH ₃ -N mg/L |
|-----------------------------------|-------------|-------------|-------------|-------------|-------------|--------------------------------|----------------------------|-------------|----------------------------|
| 0 | 27.87 | 22.97 | 3.60 | 27.74 | 26.10 | 108.28 | 8.0 | 4.56 | 1.15 |
| 15 | 7.87 | 18.01 | 3.00 | 23.91 | 21.09 | 73.88 | | | |
| 30 | 1.38 | 14.20 | 2.38 | 20.74 | 18.71 | 57.41 | | | |
| 45 | <1.10 | 8.86 | 1.64 | 16.04 | 13.24 | 39.78 | | | |
| 60 | <1.10 | 5.63 | 0.96 | 12.34 | 8.53 | 27.46 | | | |
| 75 | <1.10 | 3.36 | 0.94 | 9.29 | 7.38 | 20.97 | | | |
| 90 | <1.10 | 1.67 | 0.73 | 5.82 | 3.81 | 12.03 | | | |
| 105 | <1.10 | <1.05 | 0.42 | 5.40 | 2.54 | 8.36 | | | |
| 120 | <1.10 | — | <0.32 | 1.66 | <1.05 | 1.66 | 7.0 | 1.2 | 6.20 |
| Dup1=t ₆₀ | <1.10 | 5.38 | 1.04 | 12.48 | 8.95 | 27.85 | | | |
| Dup2=t ₁₅ | 7.53 | 17.46 | 2.91 | 22.97 | 21.79 | 72.66 | | | |
| Spike 50/50 | 14.16 | 12.32 | 2.01 | 15.12 | 13.57 | 57.18 | | | |
| DIDEH ₂ O ^a | <1.10 | <1.05 | <0.32 | <1.15 | <1.05 | | | | |
| Detection | 1.10 | 1.05 | 0.32 | 1.15 | 1.05 | | | | |
| Limits | | | | | | | | | |

a. DIDEH₂O = distilled, deionized water.

TABLE C-17. MIXTURE RUN #2 REACTOR CONDITIONS (1,000 mg/L Na₂SO₄)

| Time min | Ambient Temperature, °C | Reactor Temperature, °C | Conductance Micromhos | Volts DC | Amps DC |
|-----------------------------------|-------------------------------|-------------------------------|--------------------------|-------------|------------|
| 0 | 26.2 | 29.2 | 1300 | 21 | 3 |
| 15 | 24.4 | 34.0 | 1450 | 20 | 3 |
| 30 | 23.0 | 38.4 | 1500 | 19 | 3 |
| 45 | 22.7 | 42.0 | 1600 | 18 | 3 |
| 60 | 23.1 | 45.0 | 1700 | 18 | 3 |
| 75 | 23.2 | 47.4 | 1750 | 17 | 3 |
| 90 | 22.6 | 49.2 | 1800 | 17 | 3 |
| 105 | 22.4 | 50.4 | 1850 | 17 | 3 |
| 120 | 22.8 | 51.8 | 1850 | 17 | 3 |
| DIDEH ₂ O ^a | | | 4 | | |

TABLE C-18. MIXTURE RUN #2 RESULTS EXPLOSIVES REMOVAL

| Time min | TNT mg/L | RDX mg/L | HMX mg/L | SEX mg/L | TAX mg/L | Σ of all Explosives mg/L | NO ₃ -N mg/L | TKN mg/L | NH ₃ -N mg/L |
|-----------------------------------|-------------|-------------|-------------|-------------|-------------|--------------------------------|----------------------------|-------------|----------------------------|
| 0 | 33.42 | 27.56 | 5.86 | 21.43 | 33.94 | 122.21 | 5.6 | 3.5 | 1.05 |
| 15 | 10.00 | 19.84 | 4.92 | 20.93 | 26.01 | 81.70 | | | |
| 30 | 1.51 | 13.53 | 3.59 | 18.41 | 18.54 | 55.58 | | | |
| 45 | <0.954 | 8.73 | 2.85 | 16.04 | 13.18 | 40.80 | | | |
| 60 | — | 5.52 | 2.24 | 14.00 | 9.29 | 31.05 | | | |
| 75 | — | 2.86 | 1.47 | 11.08 | 5.51 | 20.92 | | | |
| 90 | — | 1.39 | 1.37 | 7.73 | 3.04 | 13.53 | | | |
| 105 | — | <0.879 | 0.70 | 2.95 | 1.32 | 4.97 | | | |
| 120 | — | — | <0.360 | 1.78 | <1.188 | 1.78 | 6.5 | 10.0 | 4.80 |
| Dupl=t ₁₅ | 9.97 | 20.91 | 4.81 | 19.91 | 27.11 | 82.71 | | | |
| Dupl=t ₃₀ | 1.56 | 14.12 | 3.46 | 15.19 | 19.56 | 53.89 | | | |
| Spike 50/50 | 16.90 | 20.31 | 4.19 | 17.40 | 25.78 | 84.58 | | | |
| DIDEH ₂ O ^a | <0.954 | <0.879 | <0.360 | <1.075 | <1.188 | | | | |
| Detection Limit | 0.954 | 0.879 | 0.360 | 1.075 | 1.188 | | | | |

a. DIDEH₂O = distilled, deionized water.

TABLE C-19. DATA FROM INNOVA

| Time min | Experiment #24 | | | Experiment #25 | | |
|-------------|----------------|-------------|------------|----------------|-------------|------------|
| | TNT mg/L | Volts DC | Amps DC | TNT mg/L | Volts DC | Amps DC |
| 0 | 65.67 | 14 | 3 | 74.8 | 11 | 3 |
| 15 | | | | 40.02 | 10 | 3 |
| 30 | 5.44 | 13.5 | 3 | 32.85 | 9.4 | 3 |
| 45 | | | | 9.52 | 8.6 | 3 |
| 60 | 0 | 11 | 3 | 3.04 | 8.0 | 3 |

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| 5 | US Army Medical Research and Development Command ATTN: SGRD-RMS Fort Detrick, Frederick, MD 21701 |
| 12 | Defense Technical Information Center (DTIC) ATTN: DTIC-DDA Cameron Station Alexandria, VA 22314 |
| 1 | Commandant Academy of Health Sciences, US Army ATTN: HSHA-DCD Fort Sam Houston, TX 78234 |
| 2 | Library Technician US Army Medical Bioengineering Research and Development Laboratory ATTN: SGRD-UBZ-IL Fort Detrick, Frederick, MD 21701 |
| 1 | Commander US Army Armament Research & Development Command ATTN: DRDAR-LCM-S Dover, NJ 07801 |
| 1 | Commander US Army Toxic and Hazardous Materials Agency ATTN: DRXTH-TE-D Aberdeen Proving Ground, MD 21010 |

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